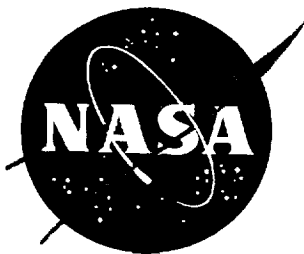


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# Microgravity Science & Applications

## Program Tasks and Bibliography for FY1994

March 1995



**National Aeronautics and  
Space Administration**

Office of Life and Microgravity  
Sciences and Applications

Microgravity Science and Applications Division

N95-28460

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H1/29 0052200

(NASA-TM-4677) MICROGRAVITY  
SCIENCE AND APPLICATIONS. PROGRAM  
TASKS AND BIBLIOGRAPHY FOR FY 1994  
(NASA) 761 p





# **Microgravity Science & Applications**

## **Program Tasks and Bibliography for FY1994**

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Washington, DC 20546-0001*

National Aeronautics and  
Space Administration  
Office of Management  
Scientific and Technical  
Information Program  
**March 1995**



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I.

Introduction

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**OBJECTIVES AND FOCUS FOR FY1994**

**NASA'S** Microgravity Science and Applications Division (MSAD) sponsors a program that seeks to focus on the use of space as a laboratory by undertaking the study of important physical, chemical, and biochemical processes on orbit that cannot easily be studied in the terrestrial gravity environment. However, since flight opportunities are rare and flight hardware is very expensive, a strong ground-based research program from which only the best experiments evolve is the keystone of the program.

The microgravity environment affords unique characteristics that allow the investigation of phenomena and processes that are difficult or impossible to study on Earth. Significant reductions in critical characteristics, such as buoyancy driven forces, convection, sedimentation, and hydrostatic pressures, make it possible to isolate and control gravity-related phenomena and make measurements that have significantly greater accuracy than can be achieved in normal gravity. Space flight gives scientists the opportunity to study the fundamental states of physical matter—solids, liquids and gases—and the forces that affect those states. Because microgravity tends to allow the treatment of gravity as a variable, research in microgravity leads to a greater fundamental understanding of the influence of gravity on the world around us. With appropriate emphasis, the results of space experiments lead to both knowledge and technological advances that have direct applications on Earth. Microgravity research also provides the practical knowledge essential to the development of future space systems.

The Office of Life and Microgravity Sciences and Applications (OLMSA) is responsible for planning and executing research stimulated by the Agency's broad scientific goals. OLMSA's Microgravity Science and Applications Division (MSAD) is responsible for guiding and focusing a comprehensive program, and currently manages its research and development tasks by dividing them into five major scientific disciplines: benchmark science, biotechnology, combustion science, fluid physics, and materials science.

Fiscal year 1994 was an important year for microgravity science in general and for MSAD in particular. Not only was the ground research program enhanced, several new investigators and research areas were added to the program through maturation of experiments in a flight development phase. The on-orbit research carried out in FY 1994 with two microgravity Space Shuttle missions is currently coming to fruition as the results from these missions are gathered and evaluated. Scientific data from the second United States Microgravity Payload (USMP-2), March 1994, and the second International Microgravity Laboratory (IML-2), July 1994, provided valuable insight into each of the five discipline fields in FY 1994. The processing and evaluation of these results have provided a solid basis for the planning of future microgravity missions, such as USML-2 scheduled in September 1995, thus beginning the research cycle anew.

This document, NASA Technical Memorandum 4677 [1995], The Microgravity Science and Applications Program Tasks and Bibliography for Fiscal Year 1994 (October 1993 – September 1994), includes research projects funded by the Office of Life and Microgravity Sciences and Applications, Microgravity Science and Applications Division, during that year. This document is published annually and is sent to scientists in the microgravity field, both foreign and domestic. The information provided in the Task Book is used in reports to the NASA Associate Administrator, the Office of Management and Budget, and to the United States Congress.

The Microgravity Science and Applications Division wishes to thank The Bionetics Corporation, and in particular recognizing Mr. Bill Wilcox (task review process and publications manager) and Mr. Duke Reiber (data system development) for their lead efforts in the development, compilation and publishing of this report. Gratitude is also expressed for significant data processing support at the responsible Centers for MSAD task management, recognizing: Dr. Mark Lysek and Angela Belcastro, JPL; Phyllis Golden, JSC; Mary Malone, LeRC; and Richard McConnell, MSFC.

## FY1994 PROGRAM RESEARCH TASK SUMMARY:

## Overview Information and Statistics

Total Number of Principal Investigators: .....	243
Total Number of Co-Investigators: .....	254
—	
Total Number of Bibliographic Listings: .....	942
• Proceedings Papers: .....	146
• Journal Articles: .....	370
• <u>NASA Tech Brief</u> Articles: .....	13
• Science/Technical Presentations: .....	389
• Books/Chapters: .....	24
—	
Total Number of Patents Applied for or Awarded: .....	1
—	
Number of Graduate Students Funded: .....	434
Number of Graduate Degrees Granted Based on MSAD-funded Research: .....	125
—	
Number of States with Funded Research (including District of Columbia): .....	36
—	
FY1993 Microgravity Science & Applications Budget: .....	\$188.0 Million

<i>Microgravity Science &amp; Applications Research Tasks and Types — Responsibilities by Center —</i>				
Centers, Types of Research	Ground	Flight	ATD	Center Totals
Jet Propulsion Laboratory	29	7	3	39
Johnson Space Flight Center	10	0	0	10
Langley Research Center	3	2	0	5
Lewis Research Center	129	35	5	169
Marshall Space Flight Center	62	25	4	91
Goddard Space Flight Center	0	0	1	1
<b>Research Task Totals</b>	<b>233</b>	<b>69</b>	<b>13</b>	<b>315</b>

## II. Microgravity Science & Applications Program Tasks for FY 1994

- **Flight Research Tasks**
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  - Biotechnology ..... II-23
  - Combustion Science ..... II-36
  - Fluid Physics ..... II-72
  - Materials Science ..... II-112
- **Ground Research Tasks**
  - Benchmark Science ..... II-159
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*Critical Dynamics in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Robert V. Duncan

Sandia National Labs, and University of New Mexico

CO-INVESTIGATORS:

Talso C.P. Chui

Jet Propulsion Laboratory (JPL)

U.E. Israelsson

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

The objective of the Critical Dynamics in Microgravity Experiment are as follows:

1. Measure the dependence of the normal state thermal conductivity ( $\lambda$ ) on the heat flux  $Q$ , and on the proximity of the cell endplate:

- Nonlinear transport very close to  $T_\lambda$
- $Q$  sets distance from criticality, as predicted by DRGT;
- Explore boundary suppression of fluctuations near criticality;
- $0.3 \text{ nW/cm}^2 \leq Q \leq 10 \text{ } \mu\text{W/cm}^2$  with no convection in a 2 cm cell.

2. Measure the temperature profile very near, and through, the HeI-HeII interface:

- Measure  $\Delta T_\lambda(Q)$  and explore "supercooled" HeI region as a function of small temperature and pressure changes
- Determine the boundary conditions on  $j_s$  and possibly  $\phi$ ;
- Determine if the interfacial width ( $w$ ) scales as predicted by DRGT and DS:  $w = w_0/\sqrt{Q}$ ;
- Measure the superfluid  $\Delta T$ , hence HeII vorticity.

3. Search for hysteresis in the superfluid transition under heat flux:

- Up/down reproducibility;
- Latent heat.

**TASK DESCRIPTION:**

A high-resolution, all-aluminum thermal conductivity cell containing a sample of ultra-high purity helium will be employed for these measurements. The high resolution thermometers (HRT) developed for and used successfully on the Lambda Point Experiment will also be used on DYNAMX to measure temperature gradients when a small heat current is applied.

**TASK SIGNIFICANCE:**

The improvements to the HRT's developed for the CHeX mission will also be applied to the DYNAMX HRT's. The all-aluminum construction will reduce the mass of the sample cell to reduce the degradation of the measurement resolution caused by ionizing radiation in orbit.

**PROGRESS DURING FY 1994:**

The DYNAMX project has worked to set up a cryoprobe for measuring the thermal conductivity of liquid helium very near the lambda transition. This cryoprobe will be used to demonstrate the feasibility of the measurements that were proposed for flight. Setting up the cryoprobe has involved: design and fabrication of a flight-like all-aluminum experimental cell; fabrication and assembly of the parts of the cryoprobe; installation of major components like the high resolution thermometers and the SQUIDS; and installation of the experimental cell with its heat flow paths tailored to the requirements of a thermal model. Some details of the progress made on each of these tasks will be provided below.

In addition to preparation of the hardware for the feasibility demonstrations, the DYNAMX team has also taken pains to communicate the aims of the experiment to the science community, and to establish channels of communication to the nonscience community. The educational and outreach actions taken by DYNAMX team members will also be delineated below.

### Hardware Development

Two considerations have led to an all-aluminum experimental cell for DYNAMX: Aluminum has a smaller cross-section for interaction with ionizing radiation than has copper, so the added thermometer noise experienced on the Lambda Point Experiment will be reduced significantly by using aluminum; and, the existence of both high-conductivity and low-conductivity forms of aluminum permits an all-welded construction with rings of high-conductivity Al spaced by the low-conductivity alloy. Attaching HRTs to the high-conductivity rings then allows the thermal profile to be measured along the walls with a negligible fraction of the heat flow through the metal, while nearly all the heat flows through the near-critical helium; high-conductivity aluminum is used for making these attachments, also. A rather detailed thermal model of the aluminum cell filled with helium near the lambda transition has directed the placement of the high-conductivity rings at three points along the cell sidewall. Two versions of the aluminum cell were built as the model was refined and as the range of parameters for testing became better defined. A two-dimensional thermal model of the cell has been developed which takes into account the radial heat flux created by the sidewall thermometer rings.

The cryoprobe for the ground-based measurements must provide the very stable thermal environment that allows high-resolution measurements to be performed as the lambda point is approached. Three temperature-controlled stages are placed between the cell and the 4 K liquid helium bath. To maintain a stable temperature at the cell, the heat leak between stages must be small and predictable. Thus, a very low pressure must be established around the platform to eliminate the vagaries of gaseous heat conduction between stages. With 42 vacuum seals and 65 electrical penetrations of the cold flange for passing signals, pump lines, and fill lines into the experiment space, preserving the vacuum integrity of this large volume presents a challenge. Each component of the DYNAMX cryoprobe has been successfully operated at low temperature during the past year.

The instrumentation for DYNAMX has progressed well. The screened room in which the ground-based experiments will be performed has been completed, and provides excellent protection from unwanted signals. Three high resolution thermometers (HRTs) have been assembled and installed in the cryoprobe, and a fourth HRT is ready for installation in the next phase of the experiment. Four SQUIDs are installed and operating well, so the HRTs can be monitored with high resolution. A fifth SQUID feedthrough is also installed on the cold flange should monitoring of a superconducting pressure transducer be required in the future. The lock-in amplifier, ratio transformer, and temperature controller chain of instruments for controlling the temperature of the stages has been shown to provide noise levels equivalent to less than  $10^{-6}$  K control uncertainty. The first implementation of the superconducting pressure transducer was found in testing to have shorts in the pick-up coil, so a rebuild is under way. An innovative DYNAMX-designed hydraulically-actuated valve for the sample cell has been fabricated and tested at liquid nitrogen temperatures, and will be ready for installation when required for filling the cell. This valve features a modular seat which may be detached from the valve stem assembly without hot work. This ease of removal permits the valve tip and the valve seat to be independently maintained and replaced.

### Education and Outreach

The DYNAMX team members have been proactive in relating the objectives and potential benefits of the mission to the public. By participating in open houses, fairs, university classes and competitions, the story of DYNAMX has been delivered to thousands of citizens.

DYNAMX has been directly involved in several educational endeavors. One graduate student and a postdoctoral fellow are continuing their education with DYNAMX support. The Principal Investigator taught a Physics Department graduate seminar at the University of New Mexico that was attended for 10 weeks by 15-25 graduate students and faculty members; the seminar described the basic principles of superfluidity and of the lambda

transition, and demonstrated the uniqueness of the DYNAMX investigation of the transition under nonequilibrium conditions. The PI has also delivered two seminars and one invited guest lecture at UNM to convey the intellectual excitement of DYNAMX.

Another direct outreach involvement by DYNAMX was the sponsorship of a competition to design a DYNAMX patch at a community college in St. Petersburg, Florida. The competition was advertised in the school newspaper to the 25,000-student community of St. Petersburg Junior College, with descriptions of the experiment and its objectives being part of the ad. 45 entries were received by the Art Department, and the five that they judged superior were forwarded to the DYNAMX team. The final selection was made at a meeting of the DYNAMX team at JPL in July. Award of a plaque will be made in November when the student designer visits the DYNAMX facility at the University of New Mexico, and again in December when the plaque will be presented at the Junior College in a public ceremony.

The DYNAMX team has also participated in presentations to the public at a JPL open house, and at two county fairs in the Los Angeles area. These presentations generally include a DYNAMX story board explaining the objectives and techniques of the DYNAMX experiment. Demonstrations of phase transitions and of superfluid and superconducting phenomena relate the experiment to everyday phenomena of phase changes out of equilibrium. Thousands of brochures describing DYNAMX as part of the program of low temperature microgravity experimentation were provided to interested persons attending these events. Well over 10,000 citizens attended these presentations according to estimates by the JPL Public Affairs Office.

Outreach to the scientific community has included several more actions by the DYNAMX team. All members participated in the pre-SCR review held in Washington in January. There the DYNAMX objectives and experiment plans were presented to a distinguished group of low temperature experimentalists and theorists comprising the JPL Low Temperature Science Steering Group. The resulting enthusiastic endorsement of the mission by the review panel reinvigorated the DYNAMX team; the panel's recommendations for actions to test the design of the DYNAMX experiment cell will be followed in detail. The DYNAMX experiment was also described to the wider audience of 75 low temperature scientists attending the 1994 JPL Workshop on Low Temperature Research in Microgravity. DYNAMX team members reached out to a very different group of scientists at the Twelfth Symposium on Thermophysical Properties at Boulder in June. The PI presented a paper describing the DYNAMX experiment, and team members heard of many other studies of phase transitions, both Earth-bound and in orbit. The PI also attended a meeting of scientists studying order-disorder transitions, held at the University of California at Santa Barbara in August, again describing the DYNAMX experiment in a presentation. At both of these meetings the team members profited from private discussions with many scientists, both for relating the nonequilibrium experiment to others, and learning of diverse studies that could be relevant to the performance of the DYNAMX experiment and to the interpretation of the results.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-24-04-04

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Duncan, R.V., Boyd, S., Moeur, W., Robinson, S., Akau, R., and Gianoulakis, S. Critical dynamics in microgravity. International Journal on Thermophysical Properties, in press, (1994).

#### Proceedings

Duncan, R.V., Boyd, S., Moeur, W., Robinson, S., Akau, R., and Gianoulakis, S. "Critical dynamics in microgravity." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

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**Presentations**

Duncan, R.V. "Progress and review of DYNAMX." Pre-SCR Review, Washington D.C., January 1994.

Duncan, R.V., Boyd, S., and Moeur, R. "Critical dynamics in microgravity." International Symposium on Order and Disorder, Santa Barbara CA, 1994.



### *Satellite Test of the Equivalence Principle (STEP)*

PRINCIPAL INVESTIGATOR: Prof. C. F. Everitt

Stanford University

CO-INVESTIGATORS:

P. Worden

Stanford University

#### **TASK OBJECTIVE:**

The objective of the Satellite Test of the Equivalence Principle (STEP) experiment is to investigate the foundation of gravitational theory, the equivalence of inertial and gravitational mass.

#### **TASK DESCRIPTION:**

The mission, now called Quick STEP, is a NASA led experiment with possible collaboration with the French space agency. The Quick STEP mission is a descoped M2 mission with the essential Equivalence Principle and geodesy science intact. The overall cost has been reduced from approximately \$600 m to \$150 m.

#### **TASK SIGNIFICANCE:**

The STEP experiment may be thought of as a modern version of the experiment attributed to Galileo of dropping two weights from the Leaning Tower of Pisa. Any difference in the ratio of gravitational to inertial mass causes a corresponding difference in the rate of fall. The detection of a difference would substantially alter present theories of relativity and gravitation.

#### **PROGRESS DURING FY 1994:**

Following the unsuccessful M2 study, the Satellite Test of the Equivalence Principle (STEP) proposal team splintered into two independent efforts: an M3 proposal headed by the European elements of the original STEP Study Team; and Quick STEP -- a JPL/Stanford effort emphasizing low cost and fast turn-around. This section outlines activities and achievements of the Quick STEP proposal team during fiscal year 1994.

In brief, the Quick STEP experiment is attempting to improve the current verification of the Equivalence Principle (a postulate of general relativity) by six orders of magnitude. This is accomplished by measuring the differential displacement of mass pairs as they travel around the Earth on a drag compensating satellite. The masses are coaxial cylindrical tubes suspended on superconducting magnetic bearings. Violations of the Equivalence Principle will manifest as a differential displacement between the masses.

A complete Satellite redesign (except Payload), utilizing off-the-shelf technology wherever possible, was needed to reduce the complexity and cost of the M2 approach. The Payload returned to its pre M2 six accelerometer configuration. This redesign started with the definition of a new Satellite configuration capable of being launched on an Orbital Science Taurus launch vehicle. The Taurus was selected for the strawman configuration because it presented the most constraining yet still possible scenario. The strawman configuration was finalized in November. Once established, the redesign turned toward three parallel activities: soliciting information about available spacecraft buses; analyzing the Satellite thermal/mechanical design; and defining the Payload/Spacecraft electrical interfaces. JPL lead the search for an acceptable off-the-shelf spacecraft bus. A Request for Information (RFI) went out in early December to a number of commercial aerospace companies soliciting input on a low cost, earth orbiting bus meeting Quick STEP's power management, data handling, and communications needs. Several responses came in below the allotted 25M cost. JPL also lead the work on thermal and mechanical design analyses. The mechanical analysis indicated a fairly conservative design with no major worries however, the thermal design pointed to heat transfer into the dewar through the warm electronics interface as an area needing further study. Finally, the definition of electrical interfaces and the overall system block diagram was a joint JPL/Stanford effort. These activities culminated in the baseline Quick STEP design furnished at the JPL/OSSI Cost Review in May and, in abridged format, at subsequent presentations to JPL senior management and to NASA Headquarters in June. Final result: excluding reserves, Quick STEP's overall mission cost, including launch, was less than 140M -- a fraction of

the original M2 cost without compromising the primary scientific objective.

Key technology development underway in FY94 include drag-free control studies, trapped flux work, helium motion analysis, and initial development of accelerometer fabrication facilities. With the exception of the analysis and design of an electrostatic helium confinement system done by Peter Mason at JPL, all technology development activities were undertaken by Stanford team members. Drag-free control studies were the basis for HaiPing Jins doctoral thesis. These analyses formed a comprehensive examination of the Quick STEP proportional thrust attitude control scheme leading to the definition of subsystem performance requirements, baseline thruster configuration, and the proposed Payload feedback control methodology. Rodney Torii, and Paul Worden began the fabrication and test of a flux microscope and niobium test samples needed to characterize how environmental effects correlate to trapped flux in superconductors. This work is essential to identify and understand factors pertinent to the superconducting bearing design. Computer analyses of the liquid helium surface shape under the tidal effects of Earth's gravity were performed by John McCuan largely for the GPB experiment but also with some relevance to Quick STEP. Helium motion has been identified as a potential noise source for the EP measurement. Lastly, the design and assembly of fabrication facilities needed for three dimensional photolithography used in the creation of the superconducting bearings, began. Matthew Bye is overseeing the development of these tools; work will continue well into fiscal 1995.

Other items of note for fiscal 1994: Stanfords new labs; the Science Review; CNES activities; and the new direction for fiscal 1995. Stanford allocated some much needed lab space to the Quick STEP team at the start of the fiscal year. This action represents a growing commitment on the part of the university toward making the Quick STEP proposal a reality. Some of the space requires substantial redesign and reconstruction to house the Quick STEP fabrication labs (and much of FY94 was spent on exactly that); this reconstruction effort is expected to extend into the middle of FY95. In the interim, the design, assembly and utilization of fabrication equipment will be accommodated in the existing (though somewhat crowded) Quick STEP lab. A Quick STEP science review was held in Washington in late February. The review board concluded that Quick STEP will: strengthen the foundation of general relativity; provide drastic improvement on the bounds of new interactions between light elementary particles; give substantial improvement in understanding the Earth's gravity field. The board also agreed that Quick STEP is the only realistic means to achieve such a large improvement in the accuracy of the verification of the Equivalence Principle, and that Quick STEP is feasible with only minor improvement in the current technology. Participation of the French Space Agency CNES has been (and still is) under consideration as a possible risk/cost/technology sharing partner with NASA in a version of Quick STEP dubbed GeoSTEP. Details of such a scenario have remained fairly fluid. In closing, Quick STEP suffered a bit of a set back at the end of fiscal 1994 when its sponsor, Microgravity (Code U), elected to delay startup due to 1995 funding realities. While this delay is disappointing, it has allowed Quick STEP time to focus on technology development and pursue a CNES and/or other outside partnership.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	1	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	3	PhD Degrees:	1

TASK INITIATION: 10/84    EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-24-07-10

RESPONSIBLE CENTER: JPL

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Swartwout, M.A., and Worden, P.W. "Error analysis for the QuickSTEP." Proc. Seventh Marcel Grossman Meeting on General Relativity, Stanford University, 1994.

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*Critical Dynamics of Fluids*

PRINCIPAL INVESTIGATOR: Prof. Richard A. Ferrell

University of Maryland

CO-INVESTIGATORS:

Dr. M. Moldover

National Institute of Standards and Technology (NIST)

Dr. R.A. Wilkinson

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

This work has a three-part objective all relating to critical phase transitions in fluids. Part one confirms the untested asymptotic limiting acoustic attenuation in simple liquid-vapor systems. The second part seeks to confirm the fast dynamics of adiabatic heat transfer in compressible liquid-vapor critical systems using a near-term space experiment. The third objective is to accelerate the theory of second sound attenuation at the lambda transition of liquid 4He. The theory would allow for comparison to data from currently planned space experiments.

**TASK DESCRIPTION:**

The work involves three critical fluid investigations: 1) ground-based definition of an acoustic attenuation in a pure liquid-vapor system space experiment, 2) Principal Investigator activities for a fast adiabatic thermal/density relaxation space experiment to fly on IML-2 in 1994, and 3) theoretical work on second sound attenuation near the lambda transition of <sup>4</sup>He. Item 1 involves repeating the Garland & Thoen experiment to establish technique and demonstrate that adiabatic heat transfer losses are detectable and predictable. That knowledge will be useful in defining a space experiment that will not be hampered by the effect. It is the space experiment that would fulfill the first objective.

Item 2 employs ESA's Critical Point Facility (CPF) with visual and interferometric data. A heat pulsed wire and a 500v charged wire are used to stimulate fast transients. This activity also allows reflight of an IML-1 experiment to study slow density relaxation with better geometric and thermal boundary conditions.

**TASK SIGNIFICANCE:**

Significance is based on the fundamental character of this work, all of which is driven by experiments only possible in low-gravity. The 4He work is to bring current theory with experiments in process. The liquid-vapor acoustics work is to probe one of the unverified predictions of older theory while properly accounting for adiabatic heat transfer loss mechanisms. The experiment on IML-2 also works toward the confirmation of the theory describing adiabatic heat transfer in highly compressible, strongly property varying, critical fluids. One and two dimensional models are still qualitative in their description of experiments.

**PROGRESS DURING FY 1994:**

I. Ambient Temperature

A. IML-2

1. TEQB: A method for projecting out the individual thermally relaxing modes of the fluid and thereby extract their specific relaxation rates, has been developed by Dr. Hong, and is currently being applied to the flight data.
2. AFEQ - heat pulse: By means of a more complete theoretical treatment, an error in the formula that has been provided by the French group has been corrected. A comparison of the corrected formula with the flight data is being carried out by Dr. Greg Zimmerli.
3. AFEQ - Voltage step: We have been able to derive an analytic expression for the electrostrictive effect as seen in the interferograms. The latter, by virtue of the microgravity environment, provide the first experimental evidence for this effect. The post-flight delivery of photographic images will soon make possible their comparison with the theoretically predicted fringe shifts.

### B. Critical Ultrasonic Attenuation

1. Theory: An analytic treatment of the corrections to dynamic scaling has been reported at the NASA workshop on low temperature physics (January 1994, Washington) and published in the proceedings. This theoretical work is also applicable to the ambient temperature phase transitions of a one component fluid.

2. Experiment: The ongoing collaboration with Dr. Bob Kusner is starting to yield data on the damping of sound waves by the effect of fast adiabatic equilibration. Visiting Professor Jayanta Bhattacharjee has determined the theoretical crossover temperature at which the bulk contribution to the attenuation becomes equal to the adiabatic surface contribution. He is in continuous contact during the course of this experiment, providing information on the method of plotting the data so as to reveal and separate these two contributions, which have a quite different physical origin.

### C. Critical Viscosity

We have reported on extended investigation into the feasibility of carrying out earth based measurements of the viscosity of a thin fluid layer closer to the critical point than has heretofore been possible.

### D. Corrections to Dynamic Scaling

An analytic treatment of this problem, as a contribution to the "Festschrift" for K. Kawasaki has been reported.

## II. Low Temperature

The method of images, combined with light scattering data, makes possible an "experimental prediction" for the Che experiment of Lipa et al.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-24-0C-25

NASA CONTRACT No.: NAG3-1395

RESPONSIBLE CENTER: LeRC

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Ferrell, R.A. and Moldover, M.R. On the limitations imposed by the Earth's gravity on measurements of the critical viscosity of a one-component fluid. *Ann. der Phys.*, (1994).

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Bhattacharjee, J.K. and Ferrell, R.A. "Corrections to dynamic scaling for the critical ultrasonic attenuation in a one-component fluid." *Proceedings of the NASA Workshop on Low-Temperature Physics*, Washington, DC, January, 1994.

Ferrell, R.A. and Bhattacharjee, J.K. "An experimental prediction for CHeX." *Proceedings of the NASA Workshop on Low-Temperature Physics*, Washington, DC, January, 1994.

*Critical Fluid Light Scattering Experiment - ZENO*

PRINCIPAL INVESTIGATOR: Prof. Robert W. Gammon

University of Maryland

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this project is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments are severely limited on Earth by the presence of gravity which causes large density gradients in the sample. The goal is to measure fluctuation decay rates with 1percent precision two decades closer to the critical point than is possible on Earth, with a temperature resolution of  $\pm 3$  microKelvin. This will require loading the sample to 0.1percent of the critical density and taking data as close as 100 microKelvin to the critical temperature ( $T_c = 289.72$  K). The minimum mission time of 100 hours will allow a complete range of temperature points to be covered, limited by the thermal response of the thermostat and correlation averaging times. Other technical problems have been addressed such as multiple scattering and the effect of wetting layers.

**TASK DESCRIPTION:**

We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), and a temperature history which can avoid wetting layers, a fast optical thermostat with microcomputer temperature control and measurement, and accurate sample loading. Further, the important engineering tasks of mounting the experiment to maintain alignment during flight have been confirmed.

The experiment entails measurement of the scattering intensity fluctuation decay rate at two angles for each temperature and simultaneously recording the scattering intensities and sample turbidity (from the transmission). The analyzed intensity and turbidity data gives the correlation length at each temperature and locates the critical temperature.

The fluctuation decay rate data set from these measurements will provide a severe test of the generalized hydrodynamics theories of transport coefficients in the critical region. When compared to equivalent data from binary liquid critical mixtures they will test the universality of critical dynamics.

**TASK SIGNIFICANCE:**

Such experiments are severely limited on Earth because gravity causes a large density gradient in the fluid due to the divergence of the fluid compressibility as the critical temperature is approached. The data from this experiment will provide a test of critical phenomena theories in a temperature realm that has not been adequately tested to date, due to the limitations imposed by gravity. The data tests the current theory of crossover from asymptotic behavior near  $T_c$  to pure background behavior far from  $T_c$ . Such a crossover theory is useful for predicting thermophysical properties in supercritical fluid solvents

**PROGRESS DURING FY 1994:**

Zeno apparatus and POCC team enjoyed a long and exciting flight on Columbia (STS-62), USMP-2 payload, in March 1994. The experiment ran for 13 days and 17 hours continuously, a probable record. The critical temperature of our xenon sample was located in orbit on a homogenous sample to 50 microK. During its run we collected 520 valid correlograms of the critical fluctuations at 35 temperatures spanning 600 mK to -100 microK from  $T_c$ . This covered the primary data range of 100 mK to 100 microK with three points per decade and added points above 100 mK in a linear steps through  $T_c$ . The average decay rate was determined from fitting all the correlograms at each temperature and gave the planned precision of 1percent.

The performance of the radiation control of the Optics Module temperature was excellent and we were able to optimize it because of the long period of holding a single Shuttle attitude. The temperature control of the sample met our stringent requirements with margin: we routinely had a less than 3 microK rms noise in three hours on our temperature readings.

We have been working toward understanding of our experiment in two ways since the mission. One has been carefully organizing the 200 Mbytes of data with some structure and indexes. Improvements were made in the speed and buffering of our ground software so that we could look at large blocks of the archival data. We have corrected an error in our analysis of the back scattering correlograms and are now approaching critical comparisons with available data such as the Berg/Moldover viscosity data set. The second major activity was to do the Tc search of our sample in extremely stable,  $\pm 10$  microK/month and to confirm that the sample had survived shipping, storage, and orbital flight, without losing more than 0.1 percent of its gas. The laser beam alignment was maintained through and after the flight.

STUDENTS FUNDED UNDER RESEARCH:				TASK INITIATION: 12/88	EXPIRATION: 2/95
BS Students:	0	BS Degrees:	0	PROJECT IDENTIFICATION: 963-50-0C-25	
MS Students:	0	MS Degrees:	0	NASA CONTRACT NO. NAS3-25370	
PhD Students:	5	PhD Degrees:	2	RESPONSIBLE CENTER: LeRC	

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*Heat Capacity Measurements Near the Lambda Point of Helium*

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PRINCIPAL INVESTIGATOR: Prof. John A. Lipa

Stanford University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

Central to condensed-matter physics is the phenomenon of second-order phase transitions. These come about when a wide class of interactive terms is added to the simple ideal gas picture of matter. To understand condensed matter in general, it is necessary to address the phase transition issue, since phase transitions are involved in nearly all interesting effects observed. Our goal is to perform the most stringent test currently feasible of the present theory of second-order phase transitions in the asymptotic limit as the transition is approached. To do this we will measure the heat capacity of helium very close to its lambda transition at 2.177 K.

**TASK DESCRIPTION:**

To perform the heat-capacity measurements, two main requirements must be met: first, we must have sufficient temperature resolution to establish the temperature scale, and second, we need to control the energy input to the sample to determine its heat capacity.

To these ends we have been developing a new high-resolution thermometer and an advanced, multilayer thermal control system. The thermometer makes use of superconducting technology to achieve a resolution of about  $3 \times 10^{-10}$  K in a 1-Hz bandwidth, and the thermal control system can achieve a power resolution approaching  $10^{-12}$  W.

**TASK SIGNIFICANCE:**

These two systems give us the capability to make measurements to the limits imposed by the Space Shuttle environment. A third requirement is to achieve an operating temperature near the lambda point. To do this we make use of the superfluid helium research facility previously flown on Spacelab-2 by JPL.

**PROGRESS DURING FY 1994:**

For the past 50 years scientists have been developing theories of condensed materials, the form of matter we are most familiar with in everyday life. These theories have had great difficulty predicting phase transitions, especially a type called co-operative transitions. Since these transitions occur in practically all forms of matter, it was a major challenge for the theories to predict them accurately. In the early '70s K. Wilson developed a new theory, based on ideas from high energy physics. This appeared to work very well and he was awarded the Nobel Prize for the work. The Lambda Point Experiment is the latest and by far the most precise test of this theory. By making measurements in microgravity it probes a transition 100 times more closely than is possible on the ground allowing substantially improved tests of the theory. The main test derived from the experiment is to compare the predicted and observed values of the exponent characterizing the divergence of the heat capacity at the transition. A second, somewhat less direct test of the theory can be made. The advantage of this test, however, is that the theoretical prediction is exact. To perform the test we must combine the heat capacity result with that from another experiment, a measurement of the behavior of the superfluid density near the transition. Theory states that the exponents from the two experiments must obey an exact relationship, called a scaling law. Fortunately, new high accuracy results have recently been obtained in this area at Stanford. Additional results from the experiment are in the area of the thermal response of the helium just above the transition. Here, measurements can be compared with the expected behavior of the thermal conductivity, based on predictions of dynamic theories of phase transitions.

This year marked the successful completion of the LPE program. All effort during the year was directed to data analysis. Numerous small effects that perturb the results were characterized and corrections applied. Unfortunately,

this process has yet to be completed in a fully satisfactory manner. The analysis is continuing on a time available basis and is expected to be complete by the end of '94. Preliminary results have been obtained in all areas. The value found for the exponent,  $-0.0128 \pm 0.0004$ , is within the current theoretical range, and five times more accurate than before. The test of the theory is weakened by the much larger uncertainties now existing in the theoretical estimates. Their uncertainties are now about 15 times greater than that in the experiment. Thus, our result will stand for some time as a benchmark for comparison with new theoretical calculations in this area. Using the scaling law and the latest Stanford superfluid density exponent, theory predicts that the heat capacity exponent should be:  $-0.0105 \pm 0.00024$ . This is off the observed value by about five standard deviations. The probability of this being a statistical accident is less than 0.1%. However, we cannot take the discrepancy too seriously just yet, because of the potential for systematic errors in the results. This is part of the reason we are proceeding very cautiously in the analysis of the flight data. The tentative result of the analysis is therefore that the theory continues to work even in the extreme conditions of the flight experiment. This gives us a great deal of confidence that Wilson's theory completely solves the phase transition problem. Other aspects of the theory, predicting the properties of condensed matter in general, need to be examined on their merits, but it is clear that the underlying principles are capable of explaining a very wide range of properties of ordinary matter.

The thermal conductivity of helium was also measured in the region just above the lambda point, where it varies very rapidly with temperature. These measurements extend the testing of phase transition dynamics by about an order of magnitude in temperature resolution. Reasonable agreement was also obtained in this area.

STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	0
PhD Students:	3	PhD Degrees:	1

TASK INITIATION: 10/86    EXPIRATION: 12/93

PROJECT IDENTIFICATION: 963-24-04-05

RESPONSIBLE CENTER: JPL

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*Confined Helium Experiment (CHeX)*

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PRINCIPAL INVESTIGATOR: Prof. John A. Lipa

Stanford University

## CO-INVESTIGATORS:

T.C.P. Chui

Jet Propulsion Laboratory (JPL)

U.E. Israelsson

Jet Propulsion Laboratory (JPL)

F.M. Gasparini

State University of New York, Buffalo

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**TASK OBJECTIVE:**

Of significant current interest in the field of condensed matter is the study of crossover behavior as a bulk system is confined more and more tightly in one or more dimensions. Crossover occurs when the effect of the boundaries is significant but not dominant. An ideal way to explore this effect is to perform measurements on films of ever decreasing thickness until the lower dimensional behavior is dominant. Unfortunately, it is not possible to vary the film thickness in most cases without totally changing the sample, making it difficult to keep track of intrinsic changes in the parameters. Near the lambda transition of helium the correlation length diverges, magnifying the effects of the confinement while simultaneously decreasing the importance of substrates. This situation gives us a unique tool to look at a diverse set of conditions in a controlled way, opening a new window on the general question of finite size phenomena in condensed matter systems. For example, at 0.1C below the transition, the correlation length is on the order of a few Angstroms, whereas 10<sup>-9</sup>C below the transition the correlation length is about 0.1 mm. If we can access the temperature region very close to the transition, we will have—for the first time—a finite size system with a truly macroscopic length scale, allowing exceptional control of the effects of boundaries. This situation appears to be absolutely unique in condensed matter systems. The Confined Helium Experiment should lead to dramatically improved tests of the theory of finite size effects.

**TASK DESCRIPTION:**

We plan to measure the heat capacity of helium confined between closely spaced parallel plates and compare the results with the bulk heat capacity data obtained on the Lambda Point Experiment (LPE). The relationship between the two data sets is predicted by theories of confinement. Most of the LPE flight hardware will be reused to perform the required measurements.

**TASK SIGNIFICANCE:**

The Confined Helium Experiment should provide a much improved test of the theory of confinement and may provide a firm basis for its extension to other properties of confined materials.

**PROGRESS DURING FY 1994:**

In the past year most of the effort has been centered on developing the confinement stack for the helium, fabricating and testing of flight prototype thermometers, and setting up the environment for flight code development. Also, at JPL, the flight cryostat performance has been enhanced to better match the needs of the mission.

The spacing in the confinement stack was changed from 100 to 50 microns on recommendation of the science review panel, to allow improved measurement of any potential deviations from theoretical predictions. This precipitated a significant effort to fabricate the new geometry using silicon wafer etching technology. This has been successful on a sample basis, and testing for flight production is currently in progress. In parallel, a 100 micron stack was subjected to a qualification shake at low temperatures with poor results. This led us to perform a major redesign of the stack to calorimeter fixturing, to minimize the stresses on the silicon. A model of the new design was recently shaken successfully.

Prototype thermometers were developed to give improved performance over the LPE devices. The bandwidth of the devices was increased to 10 Hz to minimize the effect of charged particle radiation. This change also had the effect of reducing the random noise of the devices in a 1 Hz bandwidth. Their resolution was found to be about 90 pico-Kelvins with 1 second of integration, a factor of 3 improvement over LPE. For CHEX, these devices will probably become limited by acceleration noise on the Shuttle. Since the CHEX requirement was set close to the LPE observed noise, we should have margin in this area.

For flight code development, we have set up a clone of the flight electronics and connected it to a prototype instrument, and an engineering model computer. This system is now operational and code work has started. Most of the high level code will be ported from LPE, but the lower levels will be built from scratch. Two of the major changes from LPE are 100 Hz sampling capability and 1000 byte telemetry packages. Both these features have been demonstrated.

The flight cryostat was modified to reduce the heat leak into the helium tank, extending its lifetime. Also the transfer procedure was upgraded to reduce the thermal transients on the refills, allowing a lower level to be reached without impacting the experiment. This latter improvement alleviates problems with cryostat maintenance on the pad. A 12 day hold time in orbit has been achieved, comfortably accommodating the science objectives.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 12/97

PROJECT IDENTIFICATION: 963-24-04-02

RESPONSIBLE CENTER: JPL

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*Critical Fluid Thermal Equilibrium Dynamics*

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PRINCIPAL INVESTIGATOR: Dr. R. A. WilkinsonNASA Lewis Research Center (LeRC)

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## CO-INVESTIGATORS:

Dr. R. Berg

Dr. M. Moldover

Prof. R. Gammon

Prof. J. Straub

National Institute of Standards and Technology (NIST)

National Institute of Standards and Technology (NIST)

University of Maryland

Technical University of Munich, DE

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**TASK OBJECTIVE:**

The objective of this study is to examine the thermal and density relaxation with time, after a temperature perturbation, of  $\text{SF}_6$  near its liquid-vapor critical point, in a low-gravity environment.

**TASK DESCRIPTION:**

An  $\text{SF}_6$  sample was observed using interferometry, visualization, and transmission using ESA's Critical Point Facility on IML-1. One of the scientific objectives was to observe the following: large-phase domain homogenization with and without stirring of the fluid; time evolution of heat and mass transfer after a temperature step is applied; phase evolution and configuration after transition from one-phase equilibrium to a two-phase state; and the effects of stirring on a two-phase low-gravity configuration. A second objective was to quantify the mass and thermal time constant of a one-phase system under logarithmic temperature steps.

**TASK SIGNIFICANCE:**

Critical-point experiments generally depend on achieving thermal equilibrium to within a specified tolerance and on knowing how phases develop or disappear. Data from this experiment will be used to determine the practical time-scale needed to execute meaningful critical fluid space experiments. A second use of the data was to characterize the location and growth dynamics of density or phase domains within a critical sample with vanishing surface tension.

**PROGRESS DURING FY 1994:**

The funding for this activity expired at the beginning of the FY94 fiscal year. However, work continued. A draft preprint of the low-gravity results was completed in October 1993. Key observation indicated that the observed time constants of the slowest relaxation modes were factors of two and three larger than predicted, with an unusual amount of scatter in the data. Due to the difficulty explaining the results and concerns that the experiment was misleading, the paper has not been submitted for publication. A reflight in July 1994 on IML-2 shows better results with more ideal thermal boundary conditions.

Earth-gravity data from the IML-1 apparatus was also analyzed during the fiscal year. It showed an unsystematic height dependence for the relaxation time constants that only increased the scatter seen from low-g data. The analysis clarified that when the gravity vector is along the cylindrical axis of our thin disk cell, it is a poor configuration to compare with low gravity. One had to be roughly 10 mK from  $T_c$  to get fluid time constants longer than the thermostat itself, and then the time constants were nearly an order of magnitude smaller than in low-g. When the gravity vector was along a diameter of our disk shaped cell, the time constants were roughly like those in low gravity.

This work has merged into the Ferrell Adiabatic Fast Equilibration experiment activity on IML-2.

## II. MSAD Program Tasks — Flight Research

Discipline: Benchmark Science

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/89 EXPIRATION: 9/93

PROJECT IDENTIFICATION: 963-24-05-06

RESPONSIBLE CENTER: LeRC

*Protein Crystal Growth Vapor-Diffusion Flight Hardware and Facility*

PRINCIPAL INVESTIGATOR: Dr. Daniel C. Carter

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

J. Ho (X. He)

NASA Marshall Space Flight Center (MSFC)

T. Miller

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The major objectives of this research are to provide a user-friendly interface between ground-based and flight protein crystal growth hardware, increased (common) availability of flight hardware, elimination of late access requirement, and individual loading by the principal investigator.

**TASK DESCRIPTION:**

Initially, these experiments will be conducted with a small hand-held unit using human serum albumin. Subsequent tests will involve the growth of Fab 3D6, a human anti-HIV antibody. When the necessary tests and procedures have been completed with the hand-held unit, facility flight hardware will be constructed. The microgravity experiments will be conducted in two stages. In the first stage, the hand-held unit will be flown to test the overall concept, refine the hardware if necessary, and establish protocols for later scale-ups with multi-user hardware. The design would then be configured to accommodate several trays and interface directly with the existing refrigerator/incubator module (R/IM) and thermal environment system (TES) temperature control hardware without modification. In addition, a new plastic tray will be developed to provide additional advantages in optical, storage, and hardware interfaces with a potential increase in the number of experiments in each tray assembly (VDT). In the early periods of the second stage, the facility hardware will utilize cryogenics only for improvements in experiment pre-loading efficiency. In order to proceed with the cryogenic aspects of the second stage, a sub-zero freezer will have to be developed. When flight freezers are utilized, the hardware is left in the activation configuration, and protein crystallization proceeds after the experiments are withdrawn from the freezer and placed in the temperature control units.

**TASK SIGNIFICANCE:**

This research is concerned with the development of a protein crystal growth system for microgravity which provides for rapid, convenient access to the microgravity environment and a greater number of samples, and eliminates numerous problems associated with logistics and handling of the current flight hardware.

**PROGRESS DURING FY 1994:**

1. Over the course of the past year the Hand-Held Protein Crystallization Apparatus for Microgravity (HH-PCAM) hardware was constructed, flight approved, and tested as a mid-deck hand-held experiment during STS-62. The flight contained four HH-PCAMs with a total capacity of 96 individual vapor diffusion protein crystallization chambers. The operation and crystallization experiments performed flawlessly. Some of the crystals grown were of the highest quality.
2. Based on the success of HH-PCAM as the proof of concept, facility hardware has been designed, and a prototype constructed and tested. Complete flight drawings have been approved and 18 flight units of the facility hardware constructed. The hardware, with a total of 378 individual experiments, has unprecedented capacity within the STES. Additionally, it has been designed to function in a cryogenic capacity within JSC flight dewars, as well as provide a flexible glove box interface. The cylindrical PCAMs are manifested on three upcoming flights in 1995.
3. Procedures have been established to solicit and review crystallization experiments proposed for the facility. A co-investigator group has been established.

## II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

4. Another area of the flight experiment series was initiated as a request for flight experiments which could take advantage of the longer duration, early MIR flights. The Dialysis Crystallization Apparatus for Microgravity was proposed and developed. The hardware is specifically designed to take advantage of the diffusive properties of liquids at zero-g to control inexpensively and effectively the critical approach to supersaturation. The new hardware has numerous advantages such as not requiring activation or deactivation by the crew. Current design allows 81 large volume diffusion cells which can accommodate a variety of crystallization techniques and works equally well for small molecule and protein crystallization. The hardware has great potential for a variety of ground-based applications as well.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/93 EXPIRATION: 11/97

PROJECT IDENTIFICATION: 963-23-08-08

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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*Advanced High Brilliance X-Ray Source*

PRINCIPAL INVESTIGATOR: Dr. Daniel C. Carter

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

W. Gibson

State University of New York, Albany

M. Kumakhov

I.V. Kurchatov Institute of Atomic Energy

J. Ho (X. He)

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The primary objective of this research is to produce the first x-ray generator and Kumakhov lens system optimized in design for 8.0 KeV x-rays.

**TASK DESCRIPTION:**

The x-ray unit will be integrated with existing x-ray diffraction equipment at Marshall Space Flight Center to produce a diffraction facility with the most advanced laboratory x-ray source for application in crystallography in the world. The approach to complete the task will be to produce intense small cross section parallel x-ray beams for structural analysis using third-generation Kumakhov optics.

**TASK SIGNIFICANCE:**

Protein crystallography is currently the most powerful method for the determination of the three-dimensional structure of proteins and other macromolecules. This method usually requires crystals which are relatively large in size and which possess a reasonably high degree of internal order. This research is concerned with the development of an extremely bright x-ray source for application in the evaluation and determination of the atomic structure of crystalline matter from both ground-based and current flight experiment activities.

**PROGRESS DURING FY 1994:**

The prototype x-ray lenses in this study are close packed capillary tubes with changing cross section along their entire length from input to output. An arrangement of tubes with the appropriate shape can collect and guide x-rays from a point source to produce an intense parallel beam.

1. Stage I optic completed: The first in a series of three x-ray concentrators has been completed and tested. Although not optimum in terms of transmission efficiency and focal length, this prototype optic already shows an excellent intensity increase of a factor of 34.
2. High flux microfocus source acquired and tested: A unique microfocus x-ray source was obtained from the Russian collaborators and was tested at the Center for X-ray Optics at SUNY, Albany. The measured source intensity was 30 watts at Cu Ka (8.04 KeV) in a 5  $\mu$ m spot which is at least six times greater than is available in standard commercial sources.
3. Manufacturing capability upgraded: A computerized capillary drawing fixture has been installed, replacing manual operation, and modelling has progressed to the point that actual x-ray performance can be accurately predicted. Simulation shows that an optimum shape would give enhancement of about 650 over circular collimation.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 4/93 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 963-40-20-30

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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*Protein Crystal Growth in Microgravity*

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PRINCIPAL INVESTIGATOR: Dr. Lawrence J. DeLucas

University of Alabama, Birmingham

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CO-INVESTIGATORS:

Noted Guest Investigators

See List, Appendix B

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**TASK OBJECTIVE:**

The objectives of this research are: to develop improved protein crystal growth flight hardware to produce larger, high quality protein crystals in microgravity for use in the determination of protein molecular structures for applications in medicine, drug design, agriculture, and the biological sciences; and to understand the dynamics/process of protein crystal growth.

**TASK DESCRIPTION:**

A breadboard system will be developed that utilizes light scattering to detect the onset of crystal nucleation. This optical monitoring system will be developed for both vapor diffusion and temperature-induced crystallization techniques. This hardware will be used to grow crystals with dynamic control of the appropriate crystallization parameter (i.e., temperature or vapor diffusion). The crystals will be evaluated microscopically and, from this evaluation, the best crystals will be used for x-ray data collection using the facilities available within the CMC. The data will then be compared with the best data obtained from ground-based crystals, and an evaluation of the usefulness of these dynamically-controlled systems will be made.

**TASK SIGNIFICANCE:**

Larger, high quality protein crystals may be used in molecular structure determinations for applications in medicine, drug design, agriculture, and the biological sciences.

**PROGRESS DURING FY 1994:**

- DCPCG-Vapor Diffusion

Automated dynamic control of protein crystal growth using controlled vapor diffusion has been achieved. Two primary devices have been constructed to prove the feasibility and utility of this approach. The main features of the first device include the ability to: evaporate water from a growth solution at virtually any rate, quantitatively monitor the water evaporated over time, perform up to 40 different evaporation profiles simultaneously, and use feedback from sensors to maintain or modify a given evaporation profile. This device uses a microcomputer, humidity sensors and various analog and digital interfaces under the control of custom software to effect the desired evaporation profiles. The first device has been used to show the dependence of the evaporation profile on the size and number of crystals obtained for a given solution condition. This device has proved extremely useful in showing that the crystals obtained for a given condition greatly depend upon the rate at which water is evaporated from a growth solution. The main features of the second device includes the ability to: evaporate water from a growth solution at virtually any rate, quantitatively monitor the water evaporated over time, detect and respond to nucleation events sensed by laser scattering, and perform six replicates of one user defined evaporation profile utilizing a Master/Slave configuration. This device also includes a microcomputer, a humidity sensor, various analog and digital interfaces, and custom software for controlling the system. Additionally, a laser scattering subsystem consisting of a laser, photodetector and fiber optics is used to detect nucleation events. This recently constructed device is currently being used to show the improvement that detection of nucleation and modification of the evaporation profile while the experiment is in progress can have on the crystal growth results.

### • DCPCG-Temperature

Automated dynamic control of protein crystal growth using temperature as a control parameter has been achieved. Results were presented at the 46th Annual Southeastern Regional Meeting of the American Chemical Society held in Birmingham, AL during October 16-19, 1994. The main features of the device include thermoelectric heating and cooling between about  $5-50^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ , laser scattering detection of nucleation, and user programmable temperature profiles for automated response to nucleation and subsequent growth conditions. The device has been used to prove the detection and reversibility of nucleation and the dependence of nucleation on various solution conditions such as protein concentration, pH and crystallizing agent concentration. The initial version of the device provided uniform temperature control of the entire protein solution volume. That feature proved to be useful for studying nucleation response to solution conditions, but was unsatisfactory for producing crystals with size suitable for x-ray analysis. Uniform temperature simply produces too many growth sites and a resultant shower of crystals. Second and third versions of the device are presently under study in which a localized temperature gradient (or sting) has been incorporated in an attempt to minimize the number of growth sites, thereby producing larger crystals. These systems are currently being tested; preliminary results are encouraging in that fewer but larger crystals have been grown.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	3	PhD Degrees:	0

TASK INITIATION: 4/93 EXPIRATION: 3/98

PROJECT IDENTIFICATION: 963-23-08-06

NASA CONTRACT No.NAS8-39762

RESPONSIBLE CENTER: MSFC

*Electrophoretic Separation of Cells and Particles from Rat Pituitary*

PRINCIPAL INVESTIGATOR: Dr. Wesley C. Hymer

Pennsylvania State University

CO-INVESTIGATORS:

A. Mastro

Pennsylvania State University

R. Grindeland

NASA Ames Research Center (ARC)

R. Snyder

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The objectives are to separate (1) pituitary cells and (2) hormone containing granules by free flow electrophoresis using the Japanese Free-flow Electrophoresis Unit (FFEU) on Earth and in space.

**TASK DESCRIPTION:**

To accomplish these objectives it is necessary to: (1) optimize conditions for maintaining live pituitary cells in Japanese cell culture kits (CCK) for 21 days; (2) remove cells from the CCK in space and fractionate them by electrophoresis; and (3) break open the cells in space and fractionate the lysate by electrophoresis to obtain hormone-containing granules. These procedures must be done in such a way as to be executable in flight. It is also necessary to modify existing technologies in order to analyze different hormone forms of growth hormone and prolactin in these fractions. Both hormones will be assayed by both immune and biological assays.

Because FY94 was the flight year for this experiment, the logistics associated with conducting pre-flight, flight and postflight operations was also required.

**TASK SIGNIFICANCE:**

(1) Pituitary growth hormone and prolactin are required for proper function of the bone, muscle and immune systems. Because these systems are modified by spaceflight, and because the results from 4 previous space experiments show that the biological activities of growth hormone and prolactin are diminished during and after spaceflight, this experiment is intended to probe the mechanism(s) by which these changes occur.

(2) Biotechnology research on earth routinely utilizes coupled technologies to meet focused objectives. Coupled technologies are difficult to accomplish in a low gravity environment; yet these will be routine on Space Station Alpha. This experiment serves as a prototype for such activities.

**PROGRESS DURING FY 1994:**

- (1) The flight experiment was carried out during the IML-2 mission (July, 1994).
- (2) Preliminary data document microgravity-induced changes in growth hormone cell structure and function.
- (3) Coupled technology procedures were successfully carried out in flight.
- (4) Preliminary data regarding growth hormone are to be presented at the Darmstadt, Germany meeting in November, 1994.
- (5) Active postflight analysis continues.
- (6) A repetition of the entire flight experiment on both the ground and flight FFEU hardware in Japan is being planned.

## II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/89 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-23-01-01

NASA CONTRACT No.: NAG8-953

RESPONSIBLE CENTER: MSFC

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*An Observable Protein Crystal Growth Flight Apparatus*

**PRINCIPAL INVESTIGATOR:** Dr. Alexander McPherson, Jr.

University of California, Riverside

**CO-INVESTIGATORS:**

S. Koszelak

University of California, Riverside

A. Malkin

University of California, Riverside

Y. Kuznetsov

University of California, Riverside

A. Kathman

Teledyne Brown Engineering

A. Dodds

University of California, Riverside

M. Garavito

University of Chicago

**TASK OBJECTIVE:**

The task objective is to initiate research and development efforts in the area of macromolecular crystal growth, and specifically focused on the direct observation of the relevant phenomena as they pertain to the design and ultimate flight of an observable protein crystal growth apparatus (OPCGA).

**TASK DESCRIPTION:**

The experiment objective included the identification and characterization of candidate biochemical systems that included proteins, nucleic acids, and viruses. It further included the construction of an optical platform that would be suitable for detailed interferometric analysis of protein crystal growth experiments and the visualization of concentration fields, the time lapse microphotography of macromolecular crystals, and the further characterization of the mechanisms and fundamental parameters that determine the features of macromolecular crystal growth.

**TASK SIGNIFICANCE:**

The need for structural information on biological macromolecules is of paramount importance to the emerging field of biotechnology. Such information provides the basis for the rational design of pharmaceuticals, the determination of enzymatic mechanisms and the engineering of proteins to enhance or modify their function. The value of X-ray crystallography to provide structural information at atomic resolution is unsurpassed. This technique does, however, depend on the availability of crystals for the macromolecule under study, and furthermore, which possess a high degree of internal order and suitable size and shape to allow the accurate collection of X-ray diffraction data.

Typically, crystals grown in the earth's gravitational field suffer from one or more types of flaws which decrease the structural information that can be derived from them. These imperfections include the relatively simple, but nonetheless adverse, problem of intergrowth. In a 1-g field several crystals which nucleate independently, sediment to the bottom of the growth chamber where they intergrow into a mass not suitable for X-ray diffraction analysis. Such intergrowth or the appearance of satellite crystals, which were earlier thought to occur by nucleation at the sides of defects in pre-existing crystals, have been shown to be almost exclusively due to the effects of sedimentation and inter growth. The value of a microgravity environment for the elimination of sedimentation in the preparation of these crystals is obvious. Early experiments conducted on board the space shuttle clearly indicated such benefits. They also provided the impetus for other researchers to design experiments and procedures for mimicking the effect of microgravity in ground based experiments. The development of methods for successfully growing protein crystals in gels is an example of such activities.

**PROGRESS DURING FY 1994:**

Research continued with the objective of defining the critical science requirements for design and construction of an advanced protein crystallization facility for space station, and other carriers likely to be available in the interim. This research has included time lapse video microscopy of protein crystal growth, the application of quasi elastic light scattering to prenucleation events, and more recently the introduction of both Mach-Zehnder and Michelson

interferometry. Substantial progress has been made in advancing these techniques for macromolecular crystallization. The size of the critical nucleus, surface free energy, and activation energy have been estimated for several systems that include both viruses and proteins. Interferometry has been used to visualize growth sources on growing crystal surfaces, to measure tangential and normal growth rates, and to measure the heights and slopes of growth hillocks arising from dislocations.

The International Microgravity Laboratory (IML-2) mission was successfully completed in July and the results have been analyzed by a number of techniques including X-ray diffraction. Some substantial successes in growing protein and virus crystals were achieved and a report of these results for publication is now being assembled. Most important among these were (1) the demonstration that cubic crystals of satellite tobacco mosaic virus, and both rhombohedral and hexagonal crystals of the plant seed protein canavalin, diffracted to significantly higher resolution than ground grown controls. (2) The largest particle that has been attempted was successfully crystallized for the first time. This was the plant virus Turnip Yellow Mosaic Virus (TYMV). Importantly, some distinctive alterations in the morphology of the space grown crystals were clearly evident, (3) Unique morphological changes were seen for microgravity grown crystals of hexagonal canavalin and tuna cytochrome c.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/93 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 963-23-08-07

NASA CONTRACT NO.: NAS8-3963

RESPONSIBLE CENTER: MSFC

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### *Electrophoresis Technology*

PRINCIPAL INVESTIGATOR: Dr. Robert S. Snyder

NASA Marshall Space Flight Center (MSFC)

#### CO-INVESTIGATORS:

P. Rhodes

NASA Marshall Space Flight Center (MSFC)

T. Miller

NASA Marshall Space Flight Center (MSFC)

G. Roberts

Roberts Associates, Inc.

#### TASK OBJECTIVE:

The task objectives are to study the effects of sample concentration and dielectric constant on sample stream distortion and the limits of the electrohydrodynamic stability of the sample stream in the absence of shear flow.

#### TASK DESCRIPTION:

The electrophoresis separation process can be considered to be simple in concept, but flows local to the sample filament produced by applied electric field have not been considered. These electrohydrodynamical flows, formulated by G.I. Taylor in 1965 for drops suspended in various liquids, distort the sample stream and limit the separation. In addition, electroosmosis and viscous flow, which are inherent in the continuous-flow electrophoresis device, combine to disturb further the process. Electroosmosis causes a flow in the chamber cross section which directly distorts the sample stream, which viscous flow causes a parabolic profile to develop in the flow plane. These flows distort the electrophoretic migration of samples by causing a varying residence time across the thickness of the chamber. Thus, sample constituents at the center plan will be in the electric field a different length of time and hence move more or less than comparable constituents closer to the chamber wall.

Both horizontal and vertical laboratory electrophoresis test chambers have been built to test the basic premise of continuous-flow electrophoresis that removal of buoyancy-induced thermal convection caused by axial and lateral temperature gradients will result in improved performance of these instruments in space. These gravity-dependent phenomena disturb the rectilinear flow in the separation chamber when high-voltage gradients and/or thick chambers are used, but distortion of the injected sample stream due to electrohydrodynamic effects causes major broadening of the separated bands observed in these chambers.

The initial part of the proposed space experiment was planned to be done in the French electrophoresis hardware (RAMSES) on the second International Microgravity Laboratory (IML-2). This hardware has the capability of applying the required voltage at 1,000 Hz which can permit the dielectric dependence to be determined. Two different frequencies were planned to vary the dielectric constant of the samples and the cross-section illuminator used to show the sample filament cross section, and recorded photographically. This experiment was not done on IML-2 because of a hardware failure on orbit.

The experiment can be accommodated on a later RAMSES flight, or available TEXUS electrophoresis hardware, with its cross-section illuminator, can be supplied with the required high-frequency power supply. These measurements can then be completed during a short-duration rocket flight. Additional opportunities are being evaluated.

#### TASK SIGNIFICANCE:

Since the Continuous Flow Electrophoresis System (CFES) built by the McDonnell Douglas Astronautics Company achieved results in space on seven shuttle missions that were influenced by electrohydrodynamics, these scientific phenomena are a critical part of electrophoresis in space. The severity of sample distortion due to dielectric constant variations is poorly known in the laboratory because of the concurrent sample concentration effect.



**PROGRESS DURING FY 1994:**

Most activities during FY94 were directed toward adapting our requirements to the RAMSES design for IML-2 which flew in July. The borate buffer preferred by the European Principal Investigators (PIs) was optimized for our polystyrene latex samples. A prototype RAMSES chamber was built in which we tested our latex sample stream distortions and also performed a separation of two different latex particle sizes. This chamber was also used in crew training for our portion of the RAMSES experiment.

We have completed experiments using our miniature electrohydrodynamic test chamber where we observed a conclusive dielectric effect on the sample studied. We have taken these results and compared them to the dielectric constant effect observed in drop determination of immiscible fluids. We will now quantify a selection of polystyrene latex particles with respect to Debye length, zeta potential, and particle radius. The dielectric constant of this selection can then be determined as a function of frequency by observation of sample stream distortion in our chamber.

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**STUDENTS FUNDED UNDER RESEARCH:**

TASK INITIATION: 9/89 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 963-23-08-04

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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### *Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion*

PRINCIPAL INVESTIGATOR: Prof. Robert A. Altenkirch

Mississippi State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective of this flight experiment is to determine the controlling mechanisms of flames spreading over solid fuels in the absence of buoyant or externally imposed, gas-phase flows. Ground-based testing of flame spreading in quiescent microgravity environments has identified the qualitative importance of radiative heat losses in determining flame spread rates, but these tests are too short in duration to establish spreading flames without residual effects of the ignition process.

#### TASK DESCRIPTION:

The Solid Surface Combustion Experiment (SSCE) is to be built and flown to perform a minimum of eight experiments. The experiments are to consist of five flame spreading tests using a thin fuel, varying the atmospheric composition and pressure, then three additional tests using a thick fuel with fewer variations of the same parameters. In these tests, measurements of the fuel and flame temperature are to be made and recorded, and the flames are to be photographed, using motion picture film. A parallel effort is to be made to develop a complex numerical model of the opposed-flow flame spread problem, including the important effects of surface and gas-phase radiative losses. Finally, a detailed, quantitative comparison of experimental and computed results for flame spreading over thin and thick fuels in various oxidizer and pressure environments is to be performed including comparisons of spread rate, temperature and heat transfer fields and the structure of the flame.

#### TASK SIGNIFICANCE:

The spreading of flames over solid fuels is a fundamental combustion problem that has practical significance in the prevention and control of fires. Flame spreading in normal gravity is usually dominated by buoyant air flow that introduces a significant complexity into the fundamental models of the phenomena. Experiments conducted in the microgravity environment nearly eliminate this complexity, providing a more fundamental scenario for the development of flame spreading theory.

#### PROGRESS DURING FY 1994:

During the year, the seventh flight of the Solid Surface Combustion Experiment (SSCE) was completed aboard the Shuttle Discovery during the STS-64 Mission. This flight was the second for the PMMA (polymethyl methacrylate) thick fuel phase of the flight program in which the fuel was ignited and burned in an atmosphere of 50% Oxygen/ 50% Nitrogen at a pressure of 2.0 times normal atmospheric pressure.

Other major accomplishments of the year include:

1. The results of the sixth flight of the SSCE, the first to use PMMA, included the observation that the thermocouples, near and imbedded in the fuel samples to measure temperatures, may have influenced the spreading flame by absorbing or conducting heat. A series of ground based tests was conducted to evaluate this influence, which has not been previously observed. Based on these tests the flight configuration of the thermocouples for this year's flight was changed from the original design to use much smaller thermocouple wire (1/5th the size of the original) for the gas-phase measurements. Preliminary studies of the most recent flight results show no indication of interference from the gas-phase thermocouples.

2. The two samples burned on the sixth flight of the SSCE, the first to use PMMA, showed a high degree of repeatability in the flame spreading measurement. The Principal Investigator recommended reconfiguring the software in the seventh flight so that the first sample would be quenched to obtain surface regression measurements, and the second sample would not be quenched to obtain observations of non-spreading burning after the flame reaches the end of the sample specimen. The preliminary results of the seventh flight indicate that this objective was achieved.
3. The results of the sixth flight of the SSCE indicated surface temperatures of the PMMA samples higher than are normally observed in Earth gravity experiments. The results of the recent seventh flight show more conventional temperatures and indicate the probability that the surface thermocouples in the sixth flight were uncovered by fuel pyrolysis, and hence surface regression, during the retarded flame spreading caused by the gas-phase thermocouples.
4. The results of the seventh flight of the SSCE indicate that repeatability of the experiment, from the perspective of the flame spread velocity, is again quite good. The temperature data from the two tests are of high quality and can be assembled into an integrated temperature field. These temperature data can be compared directly to temperatures computed by the numerical models developed by the Principal Investigator and can be further used to calculate heat transfer rates between the flame the fuel and the surroundings.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	8	MS Degrees:	8
PhD Students:	2	PhD Degrees:	1

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TASK INITIATION: 12/84    EXPIRATION: 12/96

PROJECT IDENTIFICATION: 963-22-05-01

RESPONSIBLE CENTER: LeRC

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Ramachandra, P.A., Altenkirch, R.A., Tang, L., Wolverton, J.K., Bhattacharjee, S. and Sacksteder, K.R. "The behavior of flames spreading over thin solids in microgravity." Twenty-Fifth Symposium (International) on Combustion, to appear in *Combustion and Flame*, 1994.

*Low-Velocity, Opposed-Flow Flame Spread in a Transport-Controlled, Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. Robert A. Altenkirch

Mississippi State University

CO-INVESTIGATORS:

S. Bhattachanjee  
S.L. Olson

San Diego State University  
NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The overall objectives of the proposed work are to uncover the underlying physics and increase the fundamental understanding of the mechanisms that cause flames to propagate over solid fuels against a low velocity flow of oxidizer in the low-gravity environment. Although the work is fundamental in nature, it has clear applications to fire safety in space and on Earth. Specific objectives are:

1. To analyze experimentally observed flame shapes, measured gas-phase field variables, spread rates, radiative characteristics, and solid-phase regression rates for comparison with theoretical prediction capability previously developed that will be continually extended.
2. To investigate the transition from ignition to either flame propagation or extinction in order to determine the characteristics of those environments that lead to flame evolution.

**TASK DESCRIPTION:**

To meet the objectives of the research program, a series of experiments has been developed to exercise several of the dimensional, controllable variables that affect the flame spread process in microgravity. Those variables that will be changed from experiment to experiment are the opposing flow velocity (1-20cm/s), the external radiant flux directed to the fuel surface (0-2) W/m<sup>2</sup>, and the oxygen concentration of the environment (35-70%). An experiment matrix is used that minimizes the number of experiments to be conducted in order to obtain the information needed to meet the scientific objectives of the effort.

Because the amount of data to be collected is limited, modelling is necessary to interpret the results and to sort out the important physics of the phenomenon. The modelling effort that will support the experimental program is numerical in nature and includes the capability to solve model flame spread problems over both thermally thin and thick fuels posed as steady-state, eigenvalue problems for spread rate or as unsteady problems from ignition through flame spread. In each approach, the two-dimensional continuity, momentum, species, and energy equations in the gas and the continuity and energy equations in the solid are solved using the SIMPLER algorithm. Gas radiation is included in the model to assist in the interpretation of species-specific emission data obtained with band pass-filtered video cameras in the experiments.

**TASK SIGNIFICANCE:**

Radiative heat transfer is critical to these and many other microgravity flame spread experiments, and so radiant heating will be imposed, and radiant heat loss will be measured. These are the first attempts at such experimental control and measurement in microgravity, and the experimental results and numerical modelling will allow the role of radiation, as well as diffusive transport, in these flames to be delineated.

**PROGRESS DURING FY 1994:**

In October 1993, Learjet-based, reduced gravity tests of the effect of the laser diode on flame spread were successfully conducted. In November 1993, the Science Requirements Document was upgraded to reflect the improved understanding of the diagnostics requirements for the experiment. Unsteady 2D flame spread computations were run with a variable low gravity level, to simulate the Learjet test conditions.

In December 1993 the Science Requirements Document was finalized. In January, DARTFire held a very successful RDR.

In February, another Learjet series was flown which revealed a problem with the laser diode system. Excellent data were obtained, however, with the intensified array camera used for flame imaging. Non-unity Lewis number capability was added to the unsteady numerical code, and work to incorporate a two-flux radiation model to the code began. The steady code was used to determine the sensitivity of the flame spread to  $g$  level. Computations showed that for  $g$  levels below  $10^{-3}$ , the effect is pretty small.

In March, the effect of variable Lewis number was examined for the base case of the DARTFire test matrix. While the unity Lewis number case extinguishes, the non-unity Lewis number propagates with significant differences in fuel vapor, water vapor, and carbon dioxide distributions. The fuel surface concentration of fuel vapor is three times that of the unity Lewis number case. Doug Seaton defended his thesis "A Complete Parametric Study of Flame Spread Over Thin Fuel in Forced-Convective and Microgravity Environment" at San Diego State University.

In April, Dr. Lin Tang visited Prof. Subrata Bhattacharjee to compare the steady and unsteady codes. Thermocouple vacuum tests were conducted to determine the effect of the laser on the thermocouple readings.

In June and July, work on reconciliation of the steady and unsteady codes was completed. 'Validation' of the unsteady thick fuel model continued. The problem of a flame spreading over a thermally thick solid is an inherently unsteady problem because of the changes of boundary layer thickness and the length of pyrolysis zone as the flame spreads. It is assumed that at any given instant, the problem can be treated as a steady problem which is the hypothesis used by Tien for a thin fuel (1979). Based on this hypothesis, a series of steady-state results was compared with the unsteady results. Good agreement was obtained for cases where the variable fields for the steady case are used as initial conditions for the unsteady case. However, for cases with an ignition transient, the flame spread rates are lower by about 30%. This is attributed to the low thermal diffusivity of the condensed phase such that the solid temperatures downstream of the flame are much lower than predicted from steady-state theory. Thus heat losses in depth are larger for the unsteady computations, and the flame propagates more slowly.

In August, DARTFire held a CDR. Computationally, the influence of gas-phase kinetic properties on ignition, flame structure and solid vaporization temperature were studied in hopes of reproducing PMMA Learjet experimental results. The predicted flame appearance agrees with the experimental one: the flame is thin, close to the fuel surface, and somewhat curved. The predicted flame moves closer to the surface when the activation energy is assumed smaller than the base case. The computational results show that the influence of gas-phase kinetic properties on surface evaporation temperature is small. The dependence of the flame spread velocity,  $V_f$ , on opposed-flow gas velocity,  $V_g$ , for thick fuels was investigated for a fully developed flow in a channel geometry. A simple formula for the spread rate was constructed as an extension of the de Ris formula and works well not only for the correct dependence of  $V_f$  on  $V_g$ , but also for predicting the flame structure. Work is now in progress on using band radiation to assist in the selection of the transmittance of the optical filters to be used in the experiments.

In September, the steady model was successfully applied to the case of "excited" flames, i.e., those that are irradiated. With an external flux of  $1 \text{ W/cm}^2$ , the spread rate increases by almost five times. Additionally, because the DARTFire geometry is, strictly speaking, a channel flow, differences between the open boundary layer flow that we have been assuming and the channel flow have been investigated. Three flames have been compared: 1) flame in an externally imposed flow, 2) flame on one side of a channel wall, and 3) flame on two walls of a channel (equivalent to solving a half-channel problem using a symmetry condition at half the channel height). Results indicate that 1) and 3) are similar in spread rate (less than 1% difference), but the velocity fields are dissimilar near the center of the channel.

The open flow produces a significantly different spread rate though because of the acceleration of the flow in the channel. Unsteady computations of channel flow have also been carried out from ignition through spread to the end of the sample. The spread rate falls from ignition and eventually approaches a relatively constant value, i.e., the same as the steady predictions. At present, we are working on including soot radiation in the radiation model and code and are checking previous work on variable properties.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	1	BS Degrees:	0
MS Students:	10	MS Degrees:	3
PhD Students:	1	PhD Degrees:	0

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TASK INITIATION: 6/91 EXPIRATION: 11/98

PROJECT IDENTIFICATION: 963-22-05-02

RESPONSIBLE CENTER: LeRC

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#### Journals

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West, J. Bhattacharjee, S. and Altenkirch, R.A. Surface radiation effects on flame spread over thermally thick fuels in an opposing flow. J. Heat Trans., 116, 646-651 (1994).

#### Presentations

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Olson, S.L., Hegde, U. "Imposed radiation effects on flame spread over black PMMA in low gravity." Fall Technical Meeting of the Eastern States Section of the Combustion Institute, December 5-7, Clearwater Florida, 1994.

West, J., Bhattacharjee, S., Rameriz, B and Altenkirch, R.A. "Low Reynolds number flow near the leading edge of a burning and non-burning plate in a microgravity environment." AIAA/ASME Thermophysics and Heat Transfer Conference, Colorado Springs, CO June 20-23, 1994.

### *Reflight of the Solid Surface Combustion Experiment with Emphasis on Flame Radiation Near Extinction*

PRINCIPAL INVESTIGATOR: Prof. Robert A. Altenkirch

Mississippi State University

#### CO-INVESTIGATORS:

Prof. S. Bhattacharjee

San Diego State University

K.R. Sacksteder

NASA Lewis Research Center (LeRC)

Dr. M. Delichatsios

Factory Mutual Research

#### TASK OBJECTIVE:

The objective of this flight experiment is to determine the mechanisms of flame spreading over solid fuels in the absence of buoyant or externally imposed, gas-phase flows. This experiment is an extension of the Solid Surface Combustion Experiment with the purpose of observing flame spread with radiative losses near the flammability limit.

#### TASK DESCRIPTION:

The Solid Surface Combustion Experiment (SSCE) is to be reflown to perform additional tests and to obtain quiescent flame spread data for cylindrical, thermally thick fuels. In these tests, measurements of the fuel and flame temperature are to be made and recorded, and the flames will be photographed using motion picture film. The numerical model, developed as part of the SSCE project, is to be further extended to predict flame spread behavior over cylindrical samples in which the geometry of the gas-phase radiative interactions are simplified computationally. A detailed quantitative comparison of the experimental and computational results is to be performed, including comparisons of spread rate, temperature field, heat transfer rates, and flame structure.

#### TASK SIGNIFICANCE:

The spreading of flames over solid fuels is a fundamental combustion problem that has practical significance in the prevention and control of fires. Flame spreading in normal gravity is usually dominated by buoyant air flow that introduces a significant complexity into fundamental theoretical models. Experiments conducted in the microgravity environment nearly eliminate this complexity, providing a more fundamental scenario for the development of flame spreading theory.

#### PROGRESS DURING FY 1994:

This is a new task in Fiscal Year 1994 beginning with the award of a new flight experiment project entitled, "Reflight of the Solid Surface Combustion Experiment with Emphasis on Flame Radiation Near Extinction," under NRA-93-OLMSA-1, "Microgravity Combustion Science: Research and Flight Experiment Opportunities."

Since the award of the new flight experiments the following accomplishments have been achieved:

1. Formulation began on the analytical treatment of flame spreading on a cylinder in a quiescent environment; this is distinct from the existing numerical modeling efforts. This treatment will include provisions for radiative losses from the flame/fuel system, leading to a model suitable for predicting flame spreading in the microgravity environment.
2. From the experimental point of view, the reflight project has been separated into two distinct parts: the first effort will be to choose an additional test condition, compatible with the existing SSCE-PMMA test matrix, but attempting to observe a flame very near the flammability limit. The second effort will be to develop a cylindrical sample to simplify the modeling effort required to simulate the gas-phase radiative losses dominating the flame spreading phenomenon in microgravity.

## II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94 EXPIRATION: 11/97

PROJECT IDENTIFICATION: 963-15-0E

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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West, J., Bhattacharjee, S., and Altenkirch, R.A. Surface radiation effects on flame spread over thermally thick fuels in an opposing flow. J. heat Trans., vol. 116, 646-651 (1994).



### *Gravitational Effects On Laminar, Transitional, and Turbulent Gas-Jet Diffusion Flames*

PRINCIPAL INVESTIGATOR: Dr. M. Y. Bahadori

Science Applications International Corporation (SAIC)

CO-INVESTIGATORS:

Dr. Uday G. Hegde

NYMA, Inc.

#### TASK OBJECTIVE:

The overall objective of this program is to improve our fundamental understanding of microgravity gas jet diffusion flames in the entire laminar, transitional, and turbulent regimes. Specifically, tests are to be conducted in microgravity with the purpose of (i) determining the effects of buoyancy on these flames, (ii) determining the relative importance of buoyancy-induced turbulence on flame characteristics, and (iii) revealing phenomena which may be masked by buoyant convection.

#### TASK DESCRIPTION:

In order to achieve these objectives, the program pursues two distinct but complementary paths, as follows:

(a) Investigate the effects of fuel type, flow rate, Reynolds number, nozzle size, and gravity on (i) global flame characteristics (such as flame shape, height, radiation and temperature), (ii) the extent of the transition regime, (iii) turbulent flame features, and (iv) stand-off characteristics and blow-off conditions. These tests will be conducted in normal-gravity and in the NASA LeRC ground-based facilities, including the Drop Tower, Zero-Gravity Facility, and/or aircraft. The measurements include temperature, thermal radiation, pressure, acceleration, and flame imaging. The data will be used to validate detailed analytical and numerical models of these flames.

(b) Identifying the mechanisms involved in the generation and interaction of observed large-scale structures which directly influence the flame characteristics noted under (a). This part involves an investigation to be carried out in space, which utilizes a controlled, well-defined set of disturbances to reveal the mechanisms that govern the dynamics of large-scale structures interacting with flame fronts under microgravity conditions. This will further our understanding of the naturally occurring disturbances that are an inherent part of the transitional and turbulent flames of part (a). As in part (a), a combined analytical and numerical modeling effort will be an integral component of this phase of the program.

#### TASK SIGNIFICANCE:

Our previous results have shown significant differences between normal gravity and microgravity flame characteristics and structure. Understanding the role of buoyancy on turbulent combustion may lead to improvements in the design of engines and furnaces.

#### PROGRESS DURING FY 1994:

Previous findings (1991-93) came from ground-based studies of high-Reynolds-number laminar, transitional, and turbulent gas jet diffusion flames in microgravity. These findings include larger microgravity turbulent flame heights and blow-off Reynolds numbers, increase in flame height with increasing Reynolds number during the transition process in microgravity, and smaller flame stand-off distances and higher blow-off Reynolds numbers for microgravity flames compared to those in normal gravity. In addition, the transition process is different in microgravity, where instabilities initiate at the base of the flame with an intermittent behavior in these naturally occurring disturbances. In contrast, the normal-gravity flames are observed to show the transition process at the tip of the flame through random instabilities.

These observations have now been confirmed through numerical studies of the flame flowfield and calculations of the critical layer for the axisymmetric mode via shear-layer instability considerations. The observed convective

velocity of the large-scale structures is in good agreement with theoretical estimates. Tests have also shown that transport processes can be influenced by the effects of buoyancy, i.e., the effective diffusivity in microgravity is smaller than in normal gravity for the transitional and turbulent flames.

The science requirements for an experiment involving forced perturbations of a laminar flame were presented during October 1993 in a Science Concept Review at the NASA Lewis Research Center. The Science Panel concluded that the proposed experiments have scientific merit, the Investigators have justified the need for microgravity experiments, and that the objectives can only be met by a space experiment that provides an extended duration of microgravity conditions. The Science Panel recommended the following: (a) Focus on axisymmetric perturbations, (b) Quantify velocity perturbations generated by the disturbance mechanism, (c) Demonstrate that the fuel will not condense in the space experiment, (d) Demonstration of adequate resolution of the imaging system, (e) Increase the sampling rate of temperature measurements, and (f) Perform a calculation to optimize the radiometer location. The following is the progress in addressing these issues:

A series of approximately 15 tests in the Zero-Gravity Facility have been identified for addressing these recommendations and to check out the flight test matrix. Analytical and numerical modeling are also underway for these efforts. In addition, since none of the recommendations has a major impact on the design of the flight experiment, design has continued in parallel with the ground-based tests. It is anticipated that the Investigators and the Engineering team will be ready for RDR in early 1995.

The design of the axisymmetric-perturbation mechanism was completed and tests showed that the components work properly to provide the required frequencies and amplitudes of oscillation. Smoke visualization tests were satisfactorily conducted at LeRC in support of the flame/ disturbance interaction characterization for flight experiment. Observations of the imposed vortex generation, convection, and interaction with the jet were made. The tests did not indicate any influence of swirl on the dynamics of the imposed vortex and subsequent entrainment into the jet, and led to a change in hardware design to reduce flow nonuniformities. In addition, LDV measurements currently in progress at Georgia Tech (through a subcontract) have shown good results for the 3-D velocity field of the vortex/jet interaction. Preliminary analysis of the data indicates that axial and radial components of velocity are indeed influenced by the vortex-generating system, whereas the tangential (i.e., swirl) component of velocity does not show this correlation.

Currently, work is underway to demonstrate that the flight camera provides adequate temporal and spatial resolution to measure the amplitude and convection velocity of the flame perturbations. Some of the verifications will be performed as part of the LeRC Zero-G Facility tests in FY95.

Measurements of the thermocouple time constant and radiometer time constant were conducted in normal gravity tests. The circuits for these measurements are being tested in LeRC's laboratories. Using established spectral analysis techniques, the temperature readings will be corrected, and tests are planned in the Zero-G Facility with the increased sampling rate. Should the ground-based tests show the need for the high sampling rate, provisions will be made to modify the flight hardware for sampling the temperature data at these rates.

Numerical calculations were performed to optimize the radiometer location for the flight experiment. Calculations for the normal-gravity flames are in good agreement with predictions of other researchers, which show that the optimum location of the radiometer is at a non-dimensional axial distance (with respect to the flame height) of 0.5-0.7. For microgravity flames, the predicted results show that the optimum location is at a non-dimensional distance of 0.3-0.5. These predictions (which demonstrate the effects of buoyancy on flame radiative characteristics) will be compared with the results of the Zero-G Facility tests for further model validation and confirmation of the optimum radiometer location for the flight experiment.

The modeling effort consists of analytical and numerical simulations of gas jet diffusion flames under the influence of imposed disturbances. To date, laminar base-flame calculations for flight experiment have been conducted. An analytical model for disturbed flames has been formulated and time-dependent flame behavior predictions are generated. Detailed numerical computations of a time-dependent, Navier-Stokes model are in progress. Disturbed

cold jets and hot jets are currently under investigation. Numerical simulation of the jet transient development under the experiment condition is obtained. Jets with both axial and radial pulsations (initial test of the model) have been studied. Detailed modeling of iris-induced flowfield in jets is in progress, and the results will be compared with the LDV measurements. Also, this comprehensive transient model is currently being extended to include the laminar flame in the presence of the iris-generated disturbances.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/91    EXPIRATION: 11/96

PROJECT IDENTIFICATION: 963-22-05-03

RESPONSIBLE CENTER: LeRC

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Hege, U.G. Heat release effects on the instability of parallel shear layers. AIAA journal, vol. 32, no. 1, 206-207 (1994).

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**Presentations**

Hege, U.G., Zhou, L., and Bahadori, M.Y. "Transitional and turbulent gas-jet diffusion flames in microgravity." Paper AIAA-94-0432, AIAA 32nd Aerospace Sciences Meeting, Reno, NV, January, 1994.

### *Sooting Effects in Reduced Gravity Droplet Combustion (SEDC)*

PRINCIPAL INVESTIGATOR: Prof. Mun Y. Choi

University of Illinois, Chicago

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

To determine the effects of sooting on droplet combustion characteristics using optical and intrusive techniques. The parameters to be studied include burning rate, flame dynamics, extinction, disruption and soot particle dynamics.

#### TASK DESCRIPTION:

The sooting behavior will be studied using expanded beam line-of sight extinction and subsequent 3-point Abel deconvolution to determine the soot volume fraction distribution; two-wavelength optical pyrometry to determine the soot temperature within the region between the flame and the droplet; and thermophoretic sampling/transmission electron microscopy to determine the soot morphology (radius of gyration, primary particle size, fractal geometry, etc). Feasibility studies will also be performed using Laser-Induced-Incandescence techniques to determine droplet soot volume fraction.

These experiments will be performed for a wide range of conditions to vary the sooting propensity of the droplet using various fuels, pressure, oxygen indices, droplet dimension and inerts as parameters. In addition, computational modeling of the soot particle dynamics using the balance between thermophoresis and Stefan drag will be compared with the experimental measurements. The modeling efforts will be advanced interactively with the experimental measurements of the soot volume fractions and the soot particle dimensions.

#### TASK SIGNIFICANCE:

The combustion of a pure, single-component liquid droplet provides an ideal problem from which to obtain valuable information for both basic and applied scientific purposes. The importance of the isolated droplet burning process has promoted extensive experimental and theoretical investigations for more than 40 years. In terms of the practical relevance, the knowledge of individual droplet burning characteristics provides insights into some of the more complex mechanisms involved in spray combustion. Since estimated energy production through spray combustion processes accounts for more than 25% of the world's output, droplet combustion remains a viable field for continued research.

Since the pioneering microgravity droplet combustion experiments of Kumagai and coworkers back in 1957, there have been numerous theoretical, computational and experimental studies analyzing the burning characteristics of isolated droplets. However, sooting effects have typically been neglected due to the complexities involved (both experimental and computational/theoretical). However, recent microgravity experiments indicate that soot/sootshell formation affects all four of the important measurable parameters involved in droplet combustion: burning rate, flame diameter, extinction and disruption. Thus, our understanding of the burning characteristics of droplets can only be complete by considering a detailed study of the sooting behavior for a wide range of characteristic times and dimensions.

In all previous microgravity studies, the degree of sooting was estimated by visual observations of the sootshell. This study will focus on the effects of sooting on the droplet burning rate, flame dimensions, extinction and disruption by accurately measuring the soot volume fraction, temperature and soot morphology and dynamics. These measurement of the temperature and soot volume fractions will also prove beneficial in assessing the importance of radiative heat transfer.

### PROGRESS DURING FY 1994:

1. Motivations and concepts for space experiments were presented to the engineering team prior to the start of the QFD (Quality Function Deployment) process. The PI participated in initial QFD sessions at LeRC to provide input regarding the Science Requirements. The meetings yielded an initial cut at the science and engineering requirements for the flight experiments. The product of the QFD process is to identify potential high-risk features of the experiment to guide the choice of which experimental elements will be breadboarded first. The subsystems of the experiment which will be breadboarded are the data storage system, droplet deployment system, droplet size measurement system, soot volume fraction measurement system, soot temperature measurement system and thermophoretic soot sampling system.
2. The PI was at Lewis during the summer months working with Dr. Randall Vander Wal and Dr. Karen Weiland on implementing Laser-Induced Incandescence for performing soot volume fraction measurements in droplet combustion. Dr. Randall Vander Wal and the PI are considering performing LII experiments in reduced-gravity droplet combustion (on DC-9) during the summer of 1995.
3. The PI has acquired a combustion chamber from LeRC for use in normal-gravity feasibility studies to be performed at UIC. For reduced-gravity testing in the ground-based facilities, discussions were held with the DCE (Droplet Combustion Experiment) project scientist to use the DCE drop rig for some preliminary tests with the PI's proposed fuels. A droplet combustion chamber which utilizes several features of the DCE drop tower hardware is being fabricated to take advantage of the design solutions. It is anticipated that an operational rig to be used in the DC-9/2.2 sec drop tower will be ready by early summer of 1995.
4. A full-field normal-gravity experimental apparatus to perform soot volume fraction and temperature measurements was designed and implemented at UIC. Performed normal gravity soot volume fraction experiments using toluene, benzene, decane and heptane at 1 atm, 0.5 atm and 0.25 atm.
5. Developed an interactive Abel-deconvolution algorithm to determine soot temperature using two-wavelength pyrometry for full-field data (512 by 512 pixel locations). Developed image processing program (using Data Translation frame grabber boards) that can produce the extinction and emission output files for direct use in the Abel deconvolution program. Tested the Abel deconvolution program using known distributions which produced very good comparisons between actual and predicted soot volume fractions. Developed similar programs to determine temperatures from 900 nm and 1000 nm emission data. Tested various wavelength combinations (700nm/800nm, 800nm/900nm, 900nm/1000nm) to determine best combination that will produce accurate results and low detection temperatures.
6. Developed a digital image processing program to determine the radius of gyration from transmission electron micrographs that are obtained using thermophoretic sampling techniques. The students are fully trained in TEM and Selected Area Electron Diffraction Analyses.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 6/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 963-15-0B

RESPONSIBLE CENTER: LeRC

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Vander Wal, R., Dietrich, D., and Choi, M.Y. "Relative soot volume fractions in droplet combustion via laser-induced incandescence." Presented at the Eastern States Section of the Combustion Institute, Clearwater, FL, December 5-7, 1994.

*Candle Flames in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Daniel L. Dietrich

NYMA, Inc.

## CO-INVESTIGATORS:

Dr. Howard Ross  
Prof. James S. TienNASA Lewis Research Center (LeRC)  
Case Western Reserve University**TASK OBJECTIVE:**

The goal of this work is to further investigate some of the features of candle flame combustion observed during a recent small scale candle flame experiment conducted on board the space shuttle. These are specifically as follows:

1. To observe whether a steady flame can exist in a purely diffusive environment;
2. To understand from a fundamental aspect the observed near-extinction flame oscillations;
3. To examine the nature of the interactions between two candle flames.

As a secondary objective, we will use the candle as a model system (non-propagating, steady-state diffusion (non-convective) flame) to investigate buoyant scaling arguments for diffusion flames. Testing and scaling analysis to date shows that utilizing reduced pressure, enhanced oxygen environments do not produce a buoyancy-free environment, as currently published scaling arguments suggest.

**TASK DESCRIPTION:**

These objectives will be accomplished by a program consisting of the development of a comprehensive numerical model of the candle flame, ground-based testing in normal-gravity laboratories and drop towers, and another small-scale glovebox experiment.

The development of a comprehensive numerical model represents the most significant addition to the proposed program. The gas-phase model will be a modification of an existing two-dimensional code developed for flame spread over solid fuels under the guidance of one of the investigators. The initial model for the wick/liquid phase has already been developed by the principal investigator.

The ground based testing will utilize existing drop rigs and other existing experimental equipment to fully characterize the candle flame in a range of ambient conditions (i.e., atmospheric to sub-atmospheric pressures, nitrogen-oxygen, helium-oxygen ambients). The data will be imaging of the flame, thermocouple measurements, LDV and possibly PIV, all of which the investigators have experience using. The use of more advanced diagnostics will also be explored during the later phases of the program.

The space experiment will be a glovebox experiment that is similar to the existing Candle Flame in Microgravity glovebox experiment (same investigators). The minor proposed changes will be: a larger free volume, the performance of the thermocouple measurements (a capability that existed but was not used on the last experiment because scheduling conflicts), the ability to maintain a large candle separation distance and to simultaneously ignite the two candles.

**TASK SIGNIFICANCE:**

The candle flame in microgravity is a unique/model system (non-propagating, steady-state diffusion (non-convective) flame). The data from this work is expected to improve our understanding of diffusion flames on a fundamental level.

### PROGRESS DURING FY 1994:

A Case Western Reserve University graduate student, J. Konecny, began working the modeling aspect of the problem in June. The initial formulation of the computational model is complete and implementation of the computer code is nearly complete. The candle will be treated as a saturated porous material at a constant temperature which greatly simplifies the wick and coupling of the wick and gas phase. The gas phase will solve the full Navier-Stokes equations with a simplified radiation model and one step chemistry.

Drop tower tests in the 5.2 second drop tower of two candles on axis candles with simultaneous ignition were conducted. Oxygen ambients of 19, 18, 17, and 16 percent with different inter-candle separation distances were tested. The results showed no ignition of either candle at 16 percent O<sub>2</sub>, and successful ignition and burning at both 19 and 18 percent. The results are currently being analyzed.

A prototype design for a liquid candle (porous metal wick with a pure liquid fuel) was completed. This is being considered as an alternative to solid candles to simplify comparisons with the theoretical predictions. The Candle Flames project was also selected as a glovebox experiment aboard the MIR glovebox. This purpose of this project is to complete some of the work not done on the USML-1 candle flames experiment and serve as a precursor to the current experiment.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 5/94 EXPIRATION: 4/98

PROJECT IDENTIFICATION: 963-15-0B

RESPONSIBLE CENTER: LeRC

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*Investigation of Laminar Jet Diffusion Flames in Microgravity: A Paradigm for Soot Processes in Turbulent Flames*

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PRINCIPAL INVESTIGATOR: Prof. Gerard M. Faeth

University of Michigan

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The flight project is an experimental and theoretical investigation of the mechanisms of soot formation in laminar jet diffusion flames under conditions of low buoyancy.

**TASK DESCRIPTION:**

Several types of experiments have been conducted. The majority have been in normal gravity, studying the influence of gravity by varying the ambient pressure. In addition, experiments have been conducted on NASA's low gravity aircraft, (the KC-135 at JSC). The work has focussed on mapping soot volume fraction, temperature, soot particle size and gas species in a variety of hydrocarbon flames. The flight experiment will be limited to measurement of soot volume fraction, soot particle size and some temperatures.

**TASK SIGNIFICANCE:**

In most flames of practical interest, soot radiation is the dominant mode of heat transport to combustor components and a dominant mechanism for flame spread and growth. The experimental results, combined with theoretical modelling will confirm or deny the applicability of the conserved scalar formalism to soot properties in diffusion flames. These results, when combined with those of ground-based low- and normal gravity experiments will increase the current fundamental understanding of soot formation processes in both laminar and turbulent flames and increase our ability to model them. Consequently this will have a significant impact on our understanding of systems such as the spread of unwanted fires, design of jet engines and large scale boilers, among others.

**PROGRESS DURING FY 1994:**

October: Additional property measurements have been conducted along the axis of the weakly buoyant flames, concentrating on samples for soot structure and TEM analysis of the samples. Measurements of the acetylene flames were reduced in order to study mechanisms of soot nucleation, growth and oxidation; the results indicated gross soot nucleation rates that were first-order in acetylene and gross soot growth rates that were roughly second-order in acetylene. Other data is being accumulated for various fuels. Finally, the LSP experiment had a successful RDR.

November: Measurements and data analysis continued in the weakly-buoyant flames considering results for acetylene as well as other fuels. Results for acetylene were completed and a paper describing the findings was prepared for the 25th International Symposium on Combustion; these results confirmed findings that soot nucleation was roughly first-order in acetylene with a modest temperature dependence (an activation energy of roughly 23 kcal/gmol) while growth is roughly second-order in acetylene concentration with a negligible activation energy. Additionally, our findings concerning soot growth in diffusion flames are similar to existing findings in premixed flames, although our nucleation rates are smaller than results of earlier studies (but the earlier work has considerable uncertainties about soot reactive surface areas).

December: Measurements and analysis of weakly-buoyant acetylene flames continued, emphasizing soot nucleation and growth. In addition, concerns about effects of acetone contamination of acetylene were addressed (acetylene is stored in contact with acetone), finding negligible effects of acetone on our results. Our newest findings about soot nucleation support first-order behavior in acetylene but growth rates appear to be somewhat lower than the second-order behavior found last month. Developmental test results concerning laser extinction measurements by imaging at LeRC were reviewed.



**January:** Study of soot growth in weakly-buoyant acetylene flames continued, with additional measurements added to the data base. This growth data are being reduced considering (1) raw data with growth due to acetylene only, (2) raw data assuming a first-order acetylene reaction (which is conventional wisdom in the literature) and an effect of age, and (3) as a net reaction rate allowing for oxidation by the low levels of oxygen present in the soot growth region (roughly 1% by volume) using the Nagle/Strickland-Constable soot oxidation results. The last appears to be most reasonable and yields a collision efficiency for acetylene of roughly 0.4%. In addition, a test campaign on the KC-135 for LSP was attempted when the KC-135 was at NASA LeRC. The campaign was largely unsuccessful because a nearby experiment on the aircraft developed a leak and sprayed the Gas Jet rig's electronic systems with a conductive water solution.

**February:** Study of soot nucleation and growth continued, with some anomalous data remeasured and all data reanalyzed. The soot growth results continue to hold up and still are similar to results in premixed flames, although we do not detect an effect of soot age on growth that generally is observed in premixed flames. Soot nucleation has remained first order in acetylene, with a low activation energy of 33 kcal/gmol. Measurements of flame and soot structure for fuels other than acetylene were initiated.

**March:** Study of soot nucleation and growth continued in order to resolve effects of soot oxidation by oxygen, carbon dioxide and water vapor. The Nagle/Strickland-Constable expression was evaluated based on our measurements of soot oxidation rates; the expression was at least order of magnitude correct. It also was found that water vapor and carbon dioxide are important for soot oxidation in the growth region, which affects the apparent order of soot growth with respect to acetylene (tending to make it larger than the actual order with respect to acetylene). The revised results, allowing for effects of oxidation by oxygen, carbon dioxide and water vapor, indicated soot growth via acetylene collisions with a collision efficiency of roughly 0.5%. Measurements continued for a variety of fuels in diffusion flames, considering both weakly-buoyant low pressure flames and buoyant flames at atmospheric pressure. In view of the relationship between present measurements and those in premixed flames, we began assembly of a premixed flame apparatus in order to carry out new measurements in premixed flames with better definition of soot surface areas than past work. A revised SRD was prepared and sent to LeRC for review.

**April:** Measurements continued in both weakly-buoyant and buoyant flames for a variety of fuels (ethylene, ethane, propane, propylene, butadiene and butane). In view of the number of flames to be considered (six) and the number of variables to be measured (soot volume fractions, soot temperature, soot structure, gas temperature, gas velocities and species concentrations) these measurements will require several report periods to complete and analyze. Work also continued on the development of the premixed flame apparatus. Round V of the KC-135 test was undertaken yielding new results for ethylene flames that currently are being analyzed.

**May:** Data from the KC-135 test was analyzed to find the correlation between burner mass flow rates and luminous flame lengths at low gravity; it was found that the length is independent of burner diameter and pressure for a given fuel flow rate but required fuel flow rates are larger than earlier estimates for a given flame length. These results were checked using weakly-buoyant diffusion flames, indicating little effect of burner diameter and pressure on the luminous flame length, similar to the KC-135 results. Tests also were undertaken to establish imaging techniques for multiline temperature measurements; this work will continue for the next few report periods. The P.I. attended the PDR and was generally satisfied with the design.

**June:** Measurements continued in order to resolve flame and soot structure for the various flames. A portion of these results involved measurements of species concentrations for an ethane flame; these measurements were in excellent agreement with estimates based on the universal state relationships developed earlier in this laboratory. Analysis of soot growth and nucleation for weakly-buoyant acetylene and butadiene flames was completed; these results are in fair agreement with our earlier acetylene mechanism but more measurements are needed before firm conclusions can be made. Test flames similar to those of Harris and Weiner have been set up on the premixed burner and we currently are working to resolve problems of flame disturbances. Measurements of flame lengths as a function of fuel flow rate for low gravity and weakly-buoyant conditions were completed; these results confirm that larger fuel flow rates are needed for a given flame length than thought previously. Tests to evaluate multiline temperature measurements by imaging continued, emphasizing effects of improved spatial resolution (or collection f number); evaluation of the results based on extrapolation of radiation-corrected thermocouple measurements from

soot free regions was satisfactory. A more definitive evaluation will require alternative temperature measurements in the soot-containing region: such measurements based on the emission and absorption properties of carbon dioxide in the infrared are being considered.

July: Measurements to resolve flame and soot structure properties for various fuels (ethylene, ethane, propane, propylene, butadiene and butane) burning in air were completed; these results currently are being analyzed to find soot nucleation and growth rates as well as continued evaluation of universal state relationships for hydrocarbon diffusion flames. Measurements of premixed ethylene/air flames were initiated finding soot volume fractions in reasonably good agreement with earlier measurements of Harris and Wiener; these measurements will continue throughout the next report period. Direct evaluation of the multiline temperature measuring system, based on imaging, will require an instrument system capable of measuring temperatures in the infrared based on the carbon dioxide bands; this arrangement currently is being designed.

August: Data from various fuel/air diffusion flames were analyzed to find soot nucleation and growth rates; results indicate an ethylene growth channel having a collision efficiency of 1.8% which is parallel to the acetylene growth channel while nucleation rates were correlated by acetylene similar to acetylene/air flames; a paper describing these results is being prepared while analysis of state relationships continues.

September: Some gaps in data relevant to fuel/air diffusion flames were found during preparation of a paper describing these results; thus measurements were completed to rectify this problem while preparation of the paper is nearing completion. Measurements of soot volume fraction distributions in the premixed ethylene/air flames were completed; work on soot temperatures began. Analysis of soot nucleation and growth was completed in three acetylene/air flames in order to check earlier results for different soot paths and flame conditions; results did confirm our earlier results and a Brief Communication is being prepared to describe the findings. Analysis of imaging measurements of soot volume fractions obtained at NASA-LeRC was undertaken. All these activities required that work on state relationships and on the evaluation of the multiline soot temperature measuring system had to be deferred; we hope to make more progress in these areas during the next year.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 2  
PhD Students: 1

TASK INITIATION: 2/92 EXPIRATION: 2/98

PROJECT IDENTIFICATION: 963-22-05-04

RESPONSIBLE CENTER: LeRC

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Presentation Sunderland, P.B., Koylu, U.O. and Faeth, G.M. "Soot formation in hydrocarbon-fueled jet diffusion flames burning in air." 25th Symposium (International) on Combustion, The Combustion Institute, Pittsburgh, poster paper, abstract only, 1994.

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### *Unsteady Diffusion Flames: Ignition, Travel, and Burnout*

PRINCIPAL INVESTIGATOR: Dr. Frank Fendell

TRW

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### **TASK OBJECTIVE:**

The objective is to investigate the adequacy of frequently adopted, simplified mathematical models to describe the behavior of laminar flames, primarily diffusion flames, by comparison with definitive experimental data, made available by burning in a microgravity environment.

#### **TASK DESCRIPTION:**

The experiment investigates the adequacy of frequently adopted, simplified mathematical models to describe the behavior of laminar flames, primarily diffusion flames, by comparison with definitive experimental data, conveniently available from burning in a microgravity environment.

#### **TASK SIGNIFICANCE:**

To study parametric variation of stoichiometric proportion and reactant dilution at ignition to provide sufficient data to test the adequacy of simplified analyses frequently used to predict diffusion-flame behavior in different practical applications. The work is particularly pertinent for flames (and near-cold-wall quench layers) involving natural gas, hydrogen, butane, propane, or other fuels that are in the gas phase at atmospheric temperature and pressure.

#### **PROGRESS DURING FY 1994:**

Experimentally, the layout of the apparatus is close to final. Parts' lists have been generated, and are ready to be placed with TRW purchasing agents, after a final critique of the design. Conceptual design of the test chamber is completed. The design will be finalized after discussions with the parylene-film manufacturer in Goleta, CA. LeRC provided information about  $H_2O$  spectral emission characteristics, spark and hot-wire ignition electrical circuitry and power requirements on the space shuttle and station to TRW. LeRC also sent TRW the results of adiabatic flame temperature calculations, done by the Project Scientist, Dr. Suleyman Gokoglu, for different initial stoichiometric proportions and reactant dilutions to help the decisions regarding experimental design and operating conditions.

The plan is to initially segregate hydrogen diluted with argon, and oxygen diluted with helium, into two equal half-volumes within a squat rectangular-solid-configured container with isothermal, noncatalytic, impervious walls. The contents of the two half volumes are each to be at atmospheric pressure and temperature, and to have equal density, so that the "average molecular weight" for each half volume is the same. The overall contents are to be sufficiently fuel-deficient such that, if (conceptually) the initial, segregated contents of the container were mixed to form a perfectly homogeneous gaseous mixture, the adiabatic flame temperature (equilibrium burned-gas temperature) would be consistent with a vigorous diffusion flame, but not so hot that either dissociation of product species (mainly water vapor) or gaseous radiative heat transfer or the physical integrity of the container is of concern.

In practice, a thin parylene film seems a candidate material to serve as the impervious initial interface, to be perforated on command at multiple sites by the movement of an intrusive "array" of pointed prongs, at least some of the tips of which are to serve as spark electrodes. Ignition is to occur after a short interval to enable some reactant interdiffusion. The diffusion flame is to propagate into that half volume which is free of the intrusive device. The key objective is to measure the flame temperature and flame position as a function of time until depletion of the deficient reactant (hydrogen) results in extinction of the planar diffusion flame.

A manuscript entitled "Unsteady Planar Diffusion Flames: Ignition, Travel, Burnout" is to be presented in the Microgravity Combustion Session at the AIAA 33rd Aerospace Sciences Meeting in Reno, NV, in January 1995. Issues to be discussed include: (1) thin-flame modeling for equidiffusion and differing diffusivities (including peripheral calculation of when the sufficiency condition for extinction, derived from a global second-order irreversible Arrhenius-type model of the finite-rate chemical kinetics, is met); (2) results for finite-rate global chemical kinetics; (3) flame quenching and possible water-vapor condensation, in proximity to the cold lateral walls of the container; and (4) assignment of values to the dimensionless parameters arising in the parabolic boundary/initial-value problem describing the travel of a planar diffusion flame. These parameters include the Lewis-Semenov numbers for fuel vapor (hydrogen) and gaseous oxidizer (oxygen), the first and second Damkohler numbers, the isentropic coefficient, the dimensionless Arrhenius activation temperature, and the stoichiometrically adjusted mass fractions for fuel vapor and gaseous oxidizer.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/94 EXPIRATION: 7/98

PROJECT IDENTIFICATION: 963-15-00

RESPONSIBLE CENTER: LeRC

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*Fundamental Study of Smoldering Combustion in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. A. C. Fernandez-Pello

University of California, Berkeley

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The overall project objective is to increase the fundamental understanding and the prediction of smoldering combustion under normal and microgravity conditions. This in turn will help the prevention and control of smolder originated fires both, in normal gravity and in space-based environments. The specific objectives are to determine the smolder characteristics of a polymeric porous combustible material (polyurethane foam), in a quiescent or convective oxidizing environment, both at normal and micro-gravity. The final objective is a set of flight experiments in a GAS Can facility in the Orbiter's cargo bay.

**TASK DESCRIPTION:**

The project objectives are accomplished by conducting experiments on ground-based and space-based facilities, and by developing theoretical models of the process. The experiments are conducted with polyurethane foam as fuel with mixtures of oxygen/nitrogen as oxidizer. Temperature at several locations of the sample are measured with thermocouples; and the resulting temperature histories are used to obtain the smolder propagation velocity and smolder reaction temperature, as a function of the oxidizer flow velocity and oxygen concentration. The experimental results are used to verify and improve the theoretical models of smolder combustion. Experiments are also conducted to observe the potential transition from smoldering to flaming combustion, and to determine the conditions and mechanisms leading to this transition.

**TASK SIGNIFICANCE:**

Smolder is important as both a fundamental combustion problem, i.e., the propagation of a heterogeneous, non-flaming, surface combustion reaction through a porous combustible material, and as a fire safety problem, i.e., the production of toxic compounds and the potential initiation of a fire through the transition to flaming. There is a need for further understanding of the problem for better prediction and control of smolder in both normal gravity and microgravity. Furthermore, microgravity introduces additional questions about the transport of mass to and from the reaction, and the transfer of heat from the reaction, that must be resolved in order to predict smolder behavior in a space-based environment.

**PROGRESS DURING FY 1994:**

**Task 1: Ignition Study of Opposed Smoldering**

The main objective of the current research is to gain an understanding, and eventually a concrete theory, of the ignition process in the smoldering of polyurethane. The immediate information we seek from the experiments are two types of plots. The first of these is the Power vs. Time plot and the second is Minimum Energy vs Power. Comparison of these curves with similar ones produced by Kitano in Japan show a number of differences. There is an apparently strange behavior of our curves at the 115W power. Further testing must be done at this power level to ensure our results are not due to experimental error. The same is true of the 100W power level. We are also planning to do some testing at 160W and 40W. In addition, several experiments were run with no air or keeping the air flow rate constant at 0.1mm/s rather than increasing it at a given time. No conclusions are yet available from these experiments but further testing of this nature could prove very interesting. Finally, a Power vs. Time curve was produced using a theoretical approach. The problem was modelled assuming one-dimensional heat conduction in a semi-infinite medium with prescribed heat flux boundary condition.

Char is also being manufactured to be used in the next space shuttle mission. Larger char samples were sent to NASA Lewis. Experiments are being run comparing the feasibility of replacing the char insulation behind the heater with shredded FiberFrax insulation.

### Task 2: Forward Smoldering

The building of the experimental apparatus has been completed. Experiments have been started to determine the ignition characteristics. Currently, the configuration of the apparatus is as follows: the test section is of a cylindrical geometry, 12cm in diameter and 30cm in length. The quality of the samples using this method is good enough such that the use of liquid nitrogen to freeze the foam is not necessary. Attachments of flanged sections of 20cm in length have been constructed (allowing for a total length of 50cm) for use in later phases of the project. Species histories for HC, CO<sub>2</sub>, CO, and O<sub>2</sub> are obtained by gas sampling. Temperature histories are gathered using special prefabricated, fast response, probe thermocouples inserted into the centerline of the section every 2cm along its length. A nichrome wire sandwiched in-between ceramic honeycomb plates ignites the foam. The bottom of the igniter is insulated with shredded Fiberfrax. Insulation below, which is a 2 micron pore size, pours metal plate acting as a flow diffuser. Bottled, compressed air serves as oxidizer for the determination of the ignition characteristics. Later phases will involve varying the oxygen concentration. The test samples are cut from a block of foam using a core drill constructed from rolled sheet metal.

Tests have been run using varying igniter powers and air flow rates to determine their effect on smolder ignition. Initial thermocouple data has indicated the possibility of two or three separate reaction fronts. We think these are the pyrolysis front and a char oxidation front in addition to the smolder front. Experiments with 1mm/s flow velocity are almost finished. We are now moving onto running experiments at 2mm/s flow velocity. Data for the 1mm/s flow velocity is being analyzed. We are looking to distinguish the pyrolysis, smolder propagation, and char oxidation fronts.

### Task 3: 2-D Smoldering with Transition to Flaming

Work has involved the modification of a wind tunnel such that a longer mixing chamber is incorporated for better mixing of aggregate exhaust gases. Data for HC, CO, CO<sub>2</sub>, and O<sub>2</sub> species histories are still being taken. An array of 10 thermocouples have also been added at the gas/solid interface in order to confirm and investigate the possibility of transition to flaming at the fuel surface exposed to the ambient. Ever since the inclusion of a 2-D matrix of 32 thermocouples embedded within the foam, experiments seem to indicate that dramatic increases in temperature within the foam (which were previously believed to reflect actual transition to flaming) well precede visual observations of flaming phenomena. The data acquisition system and software were modified to allow for recorded temperature measurements at the solid/gas interface.

Experiments for flow velocity conditions ranging from 0m/s (natural convection) to 2.5m/s have been run incorporating shear interferometry to investigate flaming phenomena at the solid/gas interface. Analysis of the additional data will hopefully verify the validity of the two flaming regimes proposed in the Fall 1993 Western States paper.

Visual observations and thermocouple histories seem to indicate that transition to flaming occurs in the char and not at the interface. Extremely localized temperature peaks (~1200C) occur in the char, well preceding flaming at the interface. It is unclear as to whether these high temperatures peaks are the result of gas phase reactions, or very exothermic, heterogeneous surface reactions. Other avenues of experimental investigation to better analyze this phenomenon are being looked into such as, x-ray tomography, ultrasonic holography, and laser absorption imaging.

Data is still being analyzed and we are submitting an abstract to the 8th International Symposium on Transport Phenomena in Combustion, S.F., U.S.A., July, 16-20, 1995. We have explored the use of ultrasonic imaging, and the results are not too promising. The foam is opaque to sound frequencies on the order of 100kHz. Lower frequencies on the order of 20kHz are being looked into. However, lower frequencies correspond to lower spacial resolution. We are still collaborating with Prof. Miyasaka at Fukui University on this technique.

## II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	2	PhD Degrees:	0

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TASK INITIATION: 3/94 EXPIRATION: 3/97

PROJECT IDENTIFICATION: 963-22-05-05

RESPONSIBLE CENTER: LeRC

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*Ignition and the Subsequent Transition to Flame Spread in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Takashi Kashiwagi

National Institute of Standards and Technology (NIST)

CO-INVESTIGATORS:

Dr. Howard Baum

National Institute of Standards and Technology (NIST)

Dr. Kevin MacGrattan

National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE:**

The objective is to conduct radiative ignition followed by transition to flame spread over various, different combustible solid surfaces in microgravity in order to understand the transition mechanisms from the ignition to subsequent flame spread, and to determine the effects of preheating, oxygen concentration, external flow velocity, geometrical configuration, and sample materials on the transition and flame spread characteristics.

These results are needed for comparison with the numerically calculated data taking advantage of microgravity to determine their accuracy and to examine the validity of the chemical and physical processes used in the calculations.

**TASK DESCRIPTION:**

The proposed space experiments will examine the effects on ignition and transition to flame spread of physical parameters which include oxygen concentration (21–50% O<sub>2</sub>), preheated fuel temperature (ambient –100 C), fuel thickness (thin/thick), fuel type (paper/pmma), geometrical configuration (including complex three-dimensional geometry such as edges, open and closed corners), external flow velocity (0–5 cm/s), and flame conditions (flaming/smoldering).

Proposed diagnostics includes a laser diode external radiant source, a digital data acquisition system, improved color flame and char front imaging, infrared imaging of the fuel surface for a time-dependent surface temperature distribution, and color shadowgraphy for flow+flame visualization. Gas species concentration measurements of O<sub>2</sub> and CO, "Desirements" include PIV, FTIR, and 2-Color Pyrometry. Standard diagnostics include thermocouples and experiment control verifications (flow, g level, radiant flux, etc.).

**TASK SIGNIFICANCE:**

For the first time, it is possible to study the transition from a radiative ignition to flame spread in the absence of overwhelming buoyant convection. The elimination of natural convective flow in the microgravity experiment simplifies both the formulation and subsequent computation of these time-dependent, three-dimensional problems that include other complexities such as finite rate chemical kinetics in both the gas and solid.

**PROGRESS DURING FY 1994:**

There is no progress to report at this time as the project has not started yet.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/94 EXPIRATION: 11/98

PROJECT IDENTIFICATION: 963-15-OB

RESPONSIBLE CENTER: LeRC

*Studies of Premixed Laminar and Turbulent Flames at Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Paul D. Ronney

University of Southern California

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this work is to study the effects of gravity-induced buoyancy on the combustion limits of premixed gas flames. The following four subtasks have been pursued:

1. Radiation effects on premixed gas flames.
2. Flame structure and stability at low Lewis number.
3. Flame propagation and extinction in cylindrical tubes.
4. Experimental simulation of combustion processes using autocatalytic chemical reactions.

**TASK DESCRIPTION:**

For task 1, we are studying the effects of the addition of inert, radiant particles to gas mixtures to increase the absorption coefficient of the gas. This enables us to study both the optically thin and optically thick radiation regimes in a single experiment.

For task 2, we are studying flames in a variety of gas mixtures having low Lewis number (Le) in aircraft tests. The goal of these studies is to determine under what conditions, if any, the instabilities which occur in low-Le mixtures may lead to the development of stable, stationary spherical flames called "flame balls."

For task 3, we have conducted experiments at Earth gravity on flame propagation in vertical tubes of varying diameter, at varying pressures and with mixtures having varying fuels, inerts, and Le, and have measured the flame propagation rates just inside the extinction limit. Since this quantity is predicted by most relevant theoretical models, the relative importance of buoyancy, flame stretch, heat loss to the tube wall, radiation loss, etc., may be assessed.

For task 4, we have introduced the use of aqueous autocatalytic propagating chemical fronts (in particular, the arsenous acid-iodate system) for the experimental simulation of premixed combustion in nonuniform and unsteady flows. These fronts more nearly match the assumptions made by most relevant theoretical models that do gaseous flames; for example, constant density, constant thermodynamic and transport processes, and no heat losses. We have studied propagation in a Taylor-Couette (TC) flow, in the annulus between two rotating concentric cylinders, and in capillary-wave (CW) flow in a thin layer of fluid in a vibrating dish.

**TASK SIGNIFICANCE:**

It is anticipated that the results of these studies will lead to an improved understanding of the fire hazards that may exist in orbiting spacecraft and of ways to minimize these hazards. Furthermore, such studies may lead to an improved understanding of the mechanisms of combustion limits in Earth-based combustion devices, which in turn could lead to the development of cleaner and more efficient engines through the use of lean premixed combustors.

**PROGRESS DURING FY 1994:**

For task (1), a radiometer preamplifier circuit was designed, built, and evaluated on real gaseous flames. It was used on several KC-135 flights in order to measure flame radiation from flame-balls. These tests were particularly important for the hydrogen-air flames which emit only weak, but fundamentally important, thermal radiation that is difficult to detect without preamplification of the radiometer signal.

For task (2), six weeks of low-gravity flight tests were performed on NASA's KC-135 research aircraft. A large amount of engineering data was obtained; no new scientific phenomena was seen, nor were any expected because all test mixtures had been studied in previous flight experiments. Several excellent examples of the "sudden propagation" phenomena, seen in methane-oxygen-sulfur hexafluoride mixtures and thought to be caused by gas radiation, were observed. The most important lessons learned which will affect the spaceflight experiment are 1) the possibility of etching of fused silica windows during spaceflight experiments due to corrosive combustion by-products, 2) the difficulties in obtaining accurate gas mixtures based on partial pressures and measured by gas chromatography, 3) the radiometers worked as expected but for hydrogen-air mixtures preamplification is required, 4) operational confidence in the spaceflight hardware can be enhanced by checking their operation of them during evacuation, chamber filling, and chamber lighting, and 5) the Xybion cameras are able to detect the combustion phenomena, however, they can be damaged by intense chamber lighting.

In addition to the buildup of the radiometer preamplifier circuit, several other hardware improvements were made. A new partial pressure gas mixing system and algorithm was built and tested in the laboratory. An improved spark energy measurement system built around a digital oscilloscope and data processing system was developed. A computerized PC-based image processing system wave was implemented. One preliminary finding is that many of the tests run in the KC-135 have resulted in overexposed images. A workstation for conducting flame-ball modeling calculations was delivered, and a one-dimensional, unsteady flame code employing detailed chemical and transport sub-models, developed by B. Rogg at Cambridge University, was obtained. This system will form the basis of the flame-ball modeling calculations to be used for comparison with the spaceflight experimental results.

The PI participated in the first meeting of the joint US/Japan Microgravity Combustion Coordinating Group. The PI proposed to perform a set of flame-ball experiments in the Hokkaido 10 second drop facility to supplement the spaceflight test matrix.

The PI also collaborated in a study with the Aerospace Corporation regarding the use of lasers for ignition. Minimum ignition energies of methane-air mixtures at 1 atm initial pressure as a function of fuel-air ratio were measured and compared to prior spark ignition and numerical modeling. The results suggest a critical role of the size of the energy deposition region, and the potential for significantly reduced minimum ignition energies compared to classical experiments if the size of the deposition region is made sufficiently small.

For task (3), to complement his current study of combustible mixtures with low Lewis number, the PI participated in a study of flame propagation at high Lewis number in tubes with Dr. Howard Pearlman, a NRC postdoctoral research associate at NASA Lewis. Theory predicts at high Lewis number, pulsating and traveling-wave instabilities should occur. The experiments have shown spiral-wave flame fronts in addition to the predicted modes. None of these modes have been conclusively observed experimentally in previous works, probably because these experiments employed more advanced diagnostics and mixtures with higher Lewis numbers than any previous work of its type.

For task (4), plans were made for the study of chemically reacting fronts responding to hydrodynamic strain. A Taylor four-roll mill, where the strain rate is determined by the rate of rotation of the rollers has been constructed.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	9	BS Degrees:	9
MS Students:	2	MS Degrees:	2
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 3/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-22-05-06

RESPONSIBLE CENTER: LeRC

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*Ignition and Flame Spread of Liquid Fuel Pools*

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PRINCIPAL INVESTIGATOR: Dr. Howard D. Ross

NASA Lewis Research Center (LeRC)

## CO-INVESTIGATORS:

Prof. W.A. Sirignano

University of California, Irvine

Dr. F.J. Miller

Case Western Reserve University

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**TASK OBJECTIVE:**

For flame spread over liquid fuel pools, the existing literature suggests three gravitational influences: (a) liquid-phase buoyant convection, delaying ignition and assisting flame spread; (b) hydrostatic pressure variation, due to variation in the liquid pool height caused by thermocapillary-induced convection; and (c) gas-phase buoyant convection in the opposite direction to the liquid-phase motion. No current model accounts for all three influences. In fact, prior to this work, there was no ability to determine whether ignition delay times and flame spread rates would be greater or lesser in low gravity.

Flame spread over liquid fuel pools is most commonly characterized by the relationship of the initial pool temperature to the fuel's idealized flash point temperature, with four or five separate characteristic regimes having been identified. In the uniform spread regime, control has been attributed to (a) gas-phase conduction and radiation, (b) gas-phase conduction only, (c) gas-phase convection and liquid conduction, and most recently (d) liquid convection ahead of the flame. Suggestions were made that the liquid convection was owed to both buoyancy and thermocapillarity. In the pulsating regime, complicated flow structures have been observed in both the gas and liquid phases, with circulation around several centers; these flows were attributed to combined thermocapillary and buoyant effects.

**TASK DESCRIPTION:**

The approach we have taken to resolving the importance of buoyancy for these flames is (a) normal gravity experiments with advanced diagnostics, (b) microgravity experiments, and (c) numerical modeling at arbitrary gravitational levels.

**TASK SIGNIFICANCE:**

Of special interest to this work is the determination of whether, and under what conditions, pulsating spread can and will occur in microgravity in the absence of buoyant flows in both phases. One possible mechanism for pulsating spread in microgravity is if the "premixed gas diffusive burning" pulsations are due to periodicity between gas-phase conductive and liquid-phase convective control. A second possibility, which will be determined by these investigations, is whether pulsations may be induced in low gravity by the presence of slow, forced, gas-phase flow.

**PROGRESS DURING FY 1994:**

During FY1994 the foremost accomplishment was the completion of the flight hardware for the first sounding rocket flight. Early in FY1994 the final fabrication of parts was completed and assembly continued throughout the spring. Component and assembly vibration tests were successfully completed at Lewis and two full normal-gravity combustion tests were carried out in the flight hardware. In both tests the hardware performed well, though an initial switch setting prevented good science data from being obtained in the first test, and a misconnected cable and poorly focused camera plagued the second test. All of these problems were traced to lapses in procedures, and steps were taken to improve the assembly and test practices. The hardware underwent another round of vibration testing at Wallops Island, and sequence testing and spin balancing were performed. The hardware is currently underway to White Sands Missile range for a scheduled November 22nd launch, the first Lewis sounding rocket experiment in nearly 30 years.

In order to obtain 1-g data for comparison purposes, and to spare the flight hardware having to undergo multiple combustion tests, a full breadboard version of SAL was designed and constructed out of (mostly) spare parts. Using this apparatus the following tests were performed.

- Using a beam splitter, video mixer, and long focal length lenses we captured some simultaneous close-up gas and liquid phase schlieren views and visible flame views of flame spread in the uniform regime. A slight preheating of the gas was visible in the schlieren view (about 1 mm), but any liquid-phase view was generally swamped by temperature gradients caused by surface evaporation. Thus, it appears for this case that the preheating occurs extremely close to the surface and the flame leading edge (on the order of 1mm), which agrees well with theoretical modelling but not with the previous interferometry work by others.
- We conducted several tests using a Kodak Ektapro High Speed video camera to capture PIV images at 500 frames/sec. Floating particles were used to record the surface movement. The video clearly indicates liquid-phase movement ahead of the flame (several mm) even in the highly uniform spread region (1-propanol, 22-23 C). However, no evidence has been found that the liquid phase accelerates to a speed faster than the flame front.
- We successfully used the Object Tracking Workstation system to track surface particles ahead of a 1-propanol flame in the uniform regime. The analysis, which involved data taken with the high speed video camera, corroborates and significantly strengthens preliminary data taken last summer which indicate surface flow ahead of the flame. The data is presented in graphs which show flame and particle location vs. time, and the particles are clearly seen to move 3-4 mm ahead of the flame, though the flame normally does overtake them.
- For pulsating spread, the high-speed video showed that the flame never caught the flow head. We also obtained good views of the vortex forming in the bulk liquid.

We continued working on improvements to the rainbow schlieren deflectometry (RSD) technique which will benefit all projects employing this diagnostic. We completed programs to make arbitrary cartesian, symmetric, gray scale, and single color filters, as well improved the optimization program to handle filters of any size and type. A NASA TM is planned to describe the new filter generation process and programs. We also performed the first quantitative comparison of RSD derived temperatures with temperatures measured independently. This was done for a stably stratified liquid layer and showed a disagreement of 0.4°C when the schlieren data was integrated over a distance of 10 mm. Work continues to improve this.

Development of a code used to animate our numerical results was largely completed. Using these animations we were able to uncover some errors in the flame spread code, notably a nonconservation of mass whenever the grid shifted. Finding such an error would have been nearly impossible using only still images, but the animations showed it clearly in the flame motion. This error and some others have now been corrected. However, a large gas-phase recirculation cell that we also discovered, and has been traced to numerical problems at the top boundary of the domain, still remains. Fortunately, it does not seem to affect the calculated flame spread rate. Particle tracking was added to the animation code, to mimic the experimental PIV data, and mock RSD and interferometry was also performed. Excellent agreement between the calculated and measured RSD views was obtained regarding vortex size and location, while differences in the calculated and measured (by others) interferometry views remain. Some differences in the surface particle trajectories still exist between the code and the experiments as well.

The Zero Gravity Facility returned to operation in FY94 and many experiments were conducted using the old hardware with some improvements such as better flow control, and a top view video camera. The drops showed successful ignition and initial spread over 1-butanol at opposed flow rates between 10 and 20 cm/s, which is the range chosen for the sounding rocket flights. We also did a few experiments with propanol which showed flame spread with opposed flow.

A low-speed wind tunnel that allows operation between 5 and 200 cm/s was set up and flow profiles were measured for several speeds and locations. Flow visualization was also conducted with dummy fuel trays in place. This tunnel will allow testing of flame spread over liquid and solids in the presence of opposed or co-current flow at normal gravity.

## II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	2	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 2/91 EXPIRATION: 2/97

PROJECT IDENTIFICATION: 962-22-05-07

RESPONSIBLE CENTER: LeRC

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*Combustion of Solid Fuel in Very Low Speed Oxygen Streams*

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PRINCIPAL INVESTIGATOR: Prof. James S. T'ien

Case Western Reserve University

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## CO-INVESTIGATORS:

K.R. Sacksteder

NASA Lewis Research Center (LeRC)

P.V. Ferkul

National Research Council

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**TASK OBJECTIVE:**

The objectives are to:

1. Enhance the understanding of flame spreading above solid-fuel surfaces through a systematic investigation in the low-speed, forced and buoyant flow regimes.
2. Determine the mechanisms that induce flammability limits of flames over solid surfaces in low-speed flows.
3. Resolve the structure of flames spreading in low-speed flows through a characterization of the velocity, temperature and selected-species fields in their vicinity.

**TASK DESCRIPTION:**

This task consists of the development and flight of a space-based experiment, the continued pursuit of ground-based results in drop towers and aircraft experiments, and the continued development of a comprehensive predictive model of concurrent-flow flame spreading.

The flight experiment is to be accomplished in three phases. An experiment to be conducted in a Glovebox Facility is to provide insight into global flame characteristics of thin and thick fuels. In the second phase, thin fuels are to be burned as they are dispensed from a continuous supply in such a way that the flame is fixed in space relative to the test apparatus. In this way, detailed diagnostic measurements are greatly simplified and transient flame behavior can be observed as the test conditions are slowly varied. In the third phase thick fuels will be burned to address the effect of transverse heat conduction into the fuel, yet with flames spreading slowly enough that the same diagnostic measurements can be made.

The ground-based experimental program consists of observations of flame spreading in partial gravity environments (first attempted as part of this program), the ignition behavior of flames over thin and thick solid fuels in microgravity forced flows, and the implementation of temperature, velocity and species concentration measurement systems.

The model development consists of integrating a fully elliptical fluid mechanics formulation with surface and gas phase radiative interactions in a transient model that attempts to predict the behavior of the selected experimental test fuels in concurrent-flow burning.

**TASK SIGNIFICANCE:**

In normal gravity, buoyant air motion in flames is at least 20 to 30 cm/sec. Flame spreading mechanisms that are present only in lower flow velocities, such as spacecraft air-ventilation currents, cannot, therefore, be studied in normal gravity. This research seeks to contribute to fundamental combustion science, and to improve the basis for engineering improved spacecraft fire safety.



### PROGRESS DURING FY 1994:

This is a new task in Fiscal Year 1994 beginning with the award of a new flight experiment project entitled, "Combustion of Solid Fuel in Very Low Speed Oxygen Streams," under NRA-93-OLMSA-1, "Microgravity Combustion Science: Research and Flight Experiment Opportunities."

Since the transition to the flight program, this task has achieved the following major accomplishments:

1. The numerical formulation of the theoretical model of concurrent flow flame spreading has been modified to permit the simulation of purely buoyant flows. This model has been used to predict flammability limits and flame spreading behavior over a range of oxygen concentrations and gravitational acceleration levels, predicting a flammability limit in air (21% oxygen) of 0.0003g.
2. The numerical formulation of the theoretical model of concurrent flow flame spreading has been modified to simulate the gas phase radiation from flames in low speed flows.
3. The Forced Flow Flamespread Test glovebox experiment (FFFT) was successfully reviewed in a peer-reviewed Science competition, and has been manifested aboard the USMP-3 mission, and is a candidate for flight aboard the MIR Space Station. An aircraft glovebox facility was prepared for testing of the FFFT in a free-float mode, unique among glovebox experiments, in which observations were made of the ignition of both thin and thick fuel sample candidates. The final design of the FFFT was completed, including the addition of a novel imaging system for in-situ, real-time velocity measurements incorporated into the video data from the experiment.
4. Experiment technology for the newly awarded flight experiment has been developed to permit the continuous dispensing of thin fuel into an established flame in low-speed forced flows in microgravity. The device consists of rolls of 7 cm wide, thin-fuel strips edged with a conductive foil to quench the fuel edge and a small combustion tunnel through which the fuel strip is pulled. An optical device detects the burnout front of the fuel where the flame is stabilized and controls the fuel feed rate. The gas flow is based upon the successful design of the Forced Flow Flamespread Test glovebox experiment technology. This device is designed to be integrated into the existing aircraft facility which was designed originally for used in purely buoyant flow flame spread studies, providing quick, low-cost access to reduced gravity testing of this new technology, and providing quick, low-cost access to rainbow schlieren measurements of forced-flow flame spreading experiments.
5. A series of experiments were completed in the NASA KC-135 aircraft providing the first stereo (3-dimensional) photographs of microgravity combustion flames; specifically downward flame spreading experiments in partial gravity environments. This imaging technology is a candidate for the newly awarded flight experiment.
6. A new set of experiments were completed in the 5 second drop tower providing observations of concurrent flow flame spread ignition processes using the flammable sample translation device developed in this program. These tests provided a direct comparison of hot-wire ignition techniques with and without a chemical promoter, and some improved observations of the beginnings of transitions toward spreading.
7. A new formulation of traditional methods for correlating opposed flow flame spreading experimental results was derived using the results of the partial gravity flame spreading tests. This new formulation for the first time correlates reduced and microgravity results with normal gravity results, accounting for radiative losses from low-speed flames.
8. A new concept for the research of partial gravity flame spreading was identified: using the Canadian Large Motion Isolation Machine (LMIM) reprogrammed to a set point of, for example, 0.001 g instead of the conventional set point of 0.0g. This device may permit observations of flame spreading in steady, milli-g environments, which may be the transition region between high-speed and low-speed buoyant flow regimes.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	3
PhD Students:	1	PhD Degrees:	2

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TASK INITIATION: 2/94 EXPIRATION: 2/99

PROJECT IDENTIFICATION: 962-22-05-40

NASA CONTRACT NO.: NAG3-1046

RESPONSIBLE CENTER: LeRC

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### *Droplet Combustion Experiment*

PRINCIPAL INVESTIGATOR: Prof. Forman A. Williams

University of California, San Diego

CO-INVESTIGATORS:

Prof. F.L. Dryer

Princeton University

#### **TASK OBJECTIVE:**

The objective of this research is to provide scientific support (in collaboration with Professor F. Dryer of Princeton University) for a droplet burning experiment to be performed on-board a space platform. This support will include performance of theoretical analyses relevant to the experiments, execution of drop-tower experiments to acquire additional data, analysis of experimental data obtained in ground-based experiments, and identification of test conditions for experiments in space.

#### **TASK DESCRIPTION:**

The objectives stated above will be pursued through the use of the NASA LeRC 2.2-Second Drop Tower with measurements made on heptane and methanol burning in atmospheres of normal air and in diluted atmospheres and through the analysis of data from this tower, and also the 5-Second Drop Tower with these fuels as well as decane. The data will be analyzed for droplet diameter and extinction diameter by use of high-precision analysis systems. Flame diameters also will be obtained by suitable digital image analysis procedures.

The theoretical approach will employ asymptotic methods to relate observed extinction conditions to elementary rate parameters. Treatment of the data by use of theory will help identify experiments that need to be done in space. Additional experiments will also be performed to address the effects of relative droplet-gas convection on burning rates. In the theoretical part of the project, a spherically symmetric, time-dependent, finite element-based numerical model with detailed gas phase kinetics, and variable property effects will be extended to study the effects of soot formation in droplet burning. Extension of the existing one-dimensional code to two-dimensional axisymmetric geometry will be evaluated.

#### **TASK SIGNIFICANCE:**

The overall purpose of the research is to achieve fundamental advances in the science of droplet combustion. In particular, unsteady liquid and gas phenomenon, and extinction chemistry of normal alkanes and alcohols are investigated.

#### **PROGRESS DURING FY 1994:**

##### **Theoretical Studies at UC-San Diego**

Analytical modeling on the structure and extinction of quasi-steady, spherically symmetric diffusion flames around methanol droplet corresponding to two, three, and four-step reduced mechanisms have been already completed. For methanol droplets, the equilibrium of water-gas shift is very good so that extinction diameters obtained from the two and three-step analysis are almost the same. The asymptotic analysis corresponding to the four-step mechanism, obtained by relaxing the H atom steady-state assumption, gives extinction diameters 50% smaller than those of the two and three-step analyses. Comparison between UCSD asymptotic results and the numerical calculations performed at Princeton show significant disagreement with the asymptotics predicting extinction diameters roughly one order magnitude smaller. When water absorption is included and an assumption of 40% water at extinction is included, the asymptotic results agree well with the numerics for methanol burned in air at 1 atm. However, at this stage the amount of water absorption at extinction must be guessed so asymptotic analysis of time-dependent water absorption problem has been started. Also, pool burning experiments to gain better understanding of the water absorption is underway.

**Numerical and Experimental Studies at Princeton****Methanol and N-Heptane Droplet Vaporization and Combustion Modeling**

A report describing in detail the numerical modeling which was performed to develop the test envelopes for the second flight test matrix entitled, "Theoretical Basis for Estimating Test Times and Conditions for Drop Tower and Space-Based Droplet Burning Experiments with Methanol and N-Heptane" was issued as Princeton University MAE Report No. 1999.

**Numerical Modeling of the Combustion of Methanol/Water Mixture Droplets**

Extensive numerical calculations for methanol/water mixture droplets of 1-5 mm diameter in air were performed. These results will be compared with those obtained using the FSDC glove box experiments (USML-2) as well as drop tower testing which will be conducted this fall.

**Development of 2-D Droplet Combustion Model**

Progress has been made in developing a droplet combustion/vaporization finite-element model. As a test case, boundary conditions for the case of pure vaporization involving two species (methanol/water) with fixed surface temperature has been implemented into the FORTRAN program. This program is currently being debugged and is expected to run shortly.

**Other**

Extensive tests were carried out for n-heptane burning in O<sub>2</sub>/He environments at the NASA Lewis 5-second drop tower. These tests cover the proposed DCE first flight test matrix. New and important results on OH-imaging of the droplet flames were accomplished using UV cameras. The test results are being analyzed and the results will be presented at the 3rd microgravity combustion workshop.

During this reporting period a successful RDR/PDR for the Droplet Combustion Experiment (DCE) was completed in collaboration with the NASA personnel.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	3	PhD Degrees:	3

TASK INITIATION: 3/91 EXPIRATION: 3/95

PROJECT IDENTIFICATION: 963-22-05-08

RESPONSIBLE CENTER: LeRC

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### *Surface Controlled Phenomena*

PRINCIPAL INVESTIGATOR: Prof. Robert E. Apfel

Yale University

CO-INVESTIGATORS:

R.G. Holt  
Yuren Tian

Jet Propulsion Laboratory  
Yale University

#### TASK OBJECTIVE:

The goals of this research are to investigate the rheological properties of surfactant-bearing liquid drops. By comparing experimental results in the ideal environment of the Spacelab to our theory for spherical equilibrium drops, we can validate the model. We can then synthesize a generic theory which can handle arbitrary acoustic fields and static deformations, in order to have a technique for studying static and dynamic surface properties for surfactant-bearing drops which can be successfully applied in 1-g experiments.

#### TASK DESCRIPTION:

Single liquid drops are introduced into the center node of an acoustic standing wave in DPM's Near-Ambient Chamber. They are allowed to reach quiescent equilibrium. Then, shape oscillations about either a spherical or a spheroidal equilibrium shape are excited - either by a momentary increase and release of the z-axis acoustic pressure or by a periodic modulation of that pressure. The resulting oscillations are recorded on video tape and ciné film for later analysis.

Three sample materials will be investigated on USML-2: triply distilled water, water with small amounts of Triton X-100 or bovine serum albumin at five different concentrations. The additives provide contrasting time scales: Triton X-100 is a nonionic fast-sorbing surfactant while BSA is sorption-inhibited. A range of drop sizes from 4 to 12 cc will be investigated for each of concentration.

#### TASK SIGNIFICANCE:

The flight experimental data will be used to validate the theory which describes drop shape-oscillations as a function of various surface parameters. These parameters will be obtained in microgravity from drops oscillating about a spherical equilibrium shape as well as about an acoustically induced oblate shape. By comparing the differences in the natural frequency and damping constant for these oscillations, the theory and experimental techniques can be used to perform measurements of the surface properties on the ground. Theoretical development and ground-based experiments to support the microgravity work are also performed.

#### PROGRESS DURING FY 1994:

Ground-based work, primarily in preparation for the USML-2 mission has focused on assuring ourselves that a) the improved DPM will work up to expectations, b) the crew will be well trained for the mission, and c) the quality of the science and the ultimate science return will be maximized.

In preparation for USML-2, tests have been performed to determine an optimal tip configuration for drop deployment; the best of the ground based designs were tested in KC-135 flights as well. Tests have been performed on whether the flexible tubing used to transport fluids for forming drops can produce bubbles. Both levitated and suspended samples are being used to study spurious sample rotation of the type observed on USML-1; from our understanding techniques for compensation are being developed.

We have designed a computer simulation of drop dynamics that can be used in training the USML-2 crew. It has been delivered to the Payload Crew Training Complex at MSFC.

Ten papers covering the experimental, analytical, and computational studies into the rheology of surfaces covering the work done in this fiscal year will be submitted within the calendar year. Experimental measurements of surface tension from the static shape of a very small levitated drop compare well with theoretical predictions. Oscillating drop experiments analogous to those of USML-1 and USML-2 have been carried out on the ground. An automated system for reducing the video image of a drop to half a dozen parameters is now processing three frames a minute. The deformation on both static and oscillating drops due to the levitating and oscillation-driving acoustic field has been studied using a boundary-integral technique. The interaction of speakers in various configurations and the resulting torques have been studied with levitated and suspended samples.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	3	BS Degrees:	3
MS Students:	2	MS Degrees:	0
PhD Students:	3	PhD Degrees:	1

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TASK INITIATION: 2/90    EXPIRATION: 6/96

PROJECT IDENTIFICATION: 963-24-04-03

RESPONSIBLE CENTER: JPL

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### *Critical Viscosity of Xenon*

PRINCIPAL INVESTIGATOR: Dr. Robert F. Berg

National Institute of Standards and Technology (NIST)

Co-INVESTIGATORS:

Dr. Michael R. Moldover

National Institute of Standards and Technology (NIST)

#### TASK OBJECTIVE:

The objective of the experiment is to produce archival viscosity data on xenon that is closer to its liquid-vapor fluid critical point than is possible in 1-g.

#### TASK DESCRIPTION:

1. Develop a low frequency, low shear rate viscometer with  $\mu\text{K}$  temperature control near room temperature. It will be of an electrostatically driven micro-flexure design;
2. Characterize vibration isolation sufficient to approach the critical temperature to within 300  $\mu\text{K}$  while measuring viscosities to 0.5% precision;
3. Load xenon sample to within 0.3% of the critical density;
4. Choose sample geometry and do heat transfer analysis to establish expected thermal gradients and thermal equilibration times realistic for a Space Shuttle flight timeline;
5. Involve critical point dynamics theorists in data analysis before and after flight.

#### TASK SIGNIFICANCE:

The data will provide complementary results with Critical Fluid Light Scattering Experiment (CFLSE) to test the mode-coupling theory of critical phenomena and provide guidance to renormalization group theory development on dynamic critical-point fluid behavior.

#### PROGRESS DURING FY 1994:

##### Oscillator calibration:

We developed a novel, accurate calibration procedure for the oscillating screen viscometer. This procedure, which makes use of the viscometer's wide bandwidth and hydrodynamic similarity, allows the viscometer to be self-calibrating. To demonstrate the validity of this procedure we measured the oscillator's transfer function under a wide variety of conditions. We obtained data using  $\text{CO}_2$  at temperatures spanning a temperature range of 35 K and densities varying by a factor of 165, thereby encountering viscosity variations as great as 50%. In contrast the flight experiment will be performed over a temperature range of 29K and at only a single density, and the viscosity is expected to change by less than 40%. The measurements showed that the viscometer's behavior is fully consistent with the use of hydrodynamic similarity for calibration.

##### Cell loading and viscometer testing near $T_c$ :

The same cell used for the  $\text{CO}_2$  tests was later filled with xenon to within 0.2% of its critical density by use of a carefully characterized filling procedure. This is better than the required accuracy of 0.3%. The critical temperature  $T_c$  was then located to a precision of better than 1 mK by observing the appearance and disappearance of the liquid-vapor meniscus. This is better than the 10 mK precision required to choose the value of the reference resistor of the cell's temperature bridge.

Viscosity measurements taken up to 40 C with the xenon-filled cell were consistent with the measurements made with the older, high-Q oscillator. However, within 0.3 K of  $T_c$  the observed transfer function deviated from the



expected response by as much as 3%. The behavior was consistent with a "sloshing" mode of the stratified of the sample, an effect which will not be present in low gravity.

### Improvements of the cell:

We implemented several improvements to the construction of the first flight cell, delivered in September 1994. The metal gaskets were made more secure by increasing the torque on the gasket's bolts to 17 inch-pounds, by using stronger bolts with a thread lubricant, and by retightening the bolts following the thermal cycles due to vacuum bakeout and sample loading. The mechanical stress on the electrical feedthrus was reduced by use of a pliable jig to increase the precision of electrode attachment and by casting epoxy around the external pins. The stainless steel fill line was replaced by a copper fill line which could be crimped reliably.

Motivated by the observation of undesired sloshing modes between 0.5 and 2.5 Hz, we added an anti-sloshing baffle to the cell. Although these modes will not exist in low gravity, they complicate the analysis of the 1-g data. The new baffle eliminated modes above 1.1 Hz, and it reduced the amplitude of the lower-frequency modes by a factor of more than two. Its design was guided by measurements on water troughs of variable geometry as well as by extensive tests in xenon cells with and without a baffle.

### Supplemental technical support:

We collaborated with Nile Oldham of NIST's Electricity Division to develop a prototype single-board, programmable, voltage divider which met CVX's requirements for precision and stability in balancing the capacitance bridge. Although the first concept of a multiplying DAC circuit exhibited an unacceptable temperature coefficient in prototype, the second concept of a 9-bit ratio transformer was successful. The prototype's transfer function matched that of the large commercial ratio transformer presently used in the laboratory, and it contributed negligibly to the noise of the viscosity measurement.

To clarify the design of the viscometry electronics, we obtained assistance from J. Cies of Hewlett-Packard in the form of a technical report.

### Miscellaneous:

The magnitudes of the drive and detection voltages were refined to reduce nonlinear behavior of the oscillator's transfer function to 0.3%. The expected effect of a possible divergence of the dielectric constant near the critical point was shown by calculation to be negligible. The effect of residual gas during the oscillator's vacuum characterization was shown to be negligible.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/94 EXPIRATION: 1/97

PROJECT IDENTIFICATION: 963-03-0A-19

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

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#### Presentations

Berg, R.F. "Hydrodynamic similarity in an oscillating body viscometer." 12th Symposium on Thermophysical Properties, Boulder, CO, June 19-24, 1994.

*The Dynamics of Disorder-Order Transitions in Hard Sphere Colloidal Dispersions*

PRINCIPAL INVESTIGATOR: Prof. Paul M. Chaikin

Princeton University

CO-INVESTIGATORS:

Prof. W.B. Russell

Princeton University

**TASK OBJECTIVE:**

To study the disorder-order transition in a hard sphere colloidal particle system, utilizing laser light scattering to detect (1) viscosity changes as the transition from fluid to crystal to glass occurs, (2) diffusion of particles, (3) nucleation and growth of the crystals and (4) rigidity after the transition to a glass is complete.

**TASK DESCRIPTION:**

The approach has focused on simulating low-gravity by fluidizing a bed of specially prepared hard spheres (silica particles with coatings of short-chain polymers) with a counterflow of solvent. This counterflow allows an approximation of microgravity.

**TASK SIGNIFICANCE:**

Materials Research - the study of crystal formation in the microgravity environment can lead to a greater understanding of how gravity affects the formation of many kinds of materials. The focus of this experiment is to use a material which can exist in multiple phases depending on its concentration in a colloidal suspension. Laser light scattering images will provide structural information about the material as it goes through liquid, ordered crystal, and disordered glass phases.

**PROGRESS DURING FY 1994:**

Light scattering has been used in the lab to verify material structure in 1-g. Sample preparation techniques are being refined which include procedures for index matching the sample and the sample container. Various volume fractions of solvent mixture have been tested to assess which combinations will be used for flight. The main focus in 1995 will be the determination and minimization of particle polydispersity of the samples.

A preliminary Science Requirement Document (SRD) was prepared and Science Concept Review (SCR) was held in January, 1994. The investigators presented the science status and plans for the flight experiment to an independent peer review panel. The review panel recommended approval of SCR based on outstanding scientific merit of the proposed experiment. NASA HQ approved the SCR.

The SRD was updated to its near-final form based on results of the ground-based testing and recommendations of the SCR peer review panel. Requirements Definition Review was held in September, 1994. The investigators made the science presentation. The RDR was also successful and the experiment has now been selected for flight.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	0
PhD Students:	0	PhD Degrees:	1

TASK INITIATION: 4/90 EXPIRATION: 7/95

PROJECT IDENTIFICATION: 963-24-0A-29

NASA CONTRACT NO.: NAG3-1158

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

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Rutgers, M.A., Dunsmuir, J., Russel, W.B., and Chaikin, P.M. Crystalline fluidized beds. Physical Review E, (to appear).

Rutgers, M.A., Dunsmuir, J., Russel, W.B., and Chaikin, P.M. Hard sphere equation of state. Science, (submitted).

*Kinetics of Diffusional Droplet Growth*

PRINCIPAL INVESTIGATOR: Dr. Donald O. Frazier

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

M. Glicksman

Rensselaer Polytechnic Institute

J. Rogers

NASA Marshall Space Flight Center (MSFC)

J. Downey

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

To determine droplet growth rate constants and changes in size distributions using optical techniques for recording droplet data with time.

**TASK DESCRIPTION:**

A transparent model binary liquid/liquid miscibility-gap type solution was the system of choice. The experimental approach was to deploy a fixed array of droplets of one liquid phase into a cell filled with the conjugate phase. This approach was to greatly reduce any residual gravity effects and eliminate the possibility of coalescence. Ground-based studies were to determine the experimental parameters required to maximize the amount of data on "pure" Ostwald ripening obtainable during the flight experiment. For tethered droplets, it was considered important to determine such parameters as volume fraction of droplet phase, initial droplet number, droplet spacing and sizes, the time required for the experiment, and ideal concentrations for supersaturation and growth.

**TASK SIGNIFICANCE:**

To a large extent, particle growth and distribution determine the mechanical properties of an alloy. Capabilities provided by holographic techniques enabling observations of diffusional growth of a second phase in a model, transparent two-phase system, allow detailed analyses of particle size and number distribution. Direct observation of these phenomena allow experimental determination of the scaling laws which define the time evolution of discrete phase droplets in the asymptotic limit. To our knowledge, this technique is unique for providing real-time optical observation of these processes.

**PROGRESS DURING FY 1994:**

Our work has resulted in determination of the best methods for performing ground-based testing. Ground work requires a transparent isopycnic system. Sensitivity to gravitational fields should be tested, in the laboratory, by tethering two droplets in a test cell and comparing growth kinetics at varying temperatures. Convective flows increase with increasing conjugate phase density differences which are relatively strong functions of temperature. Quench experiments in narrow path-length cells should allow observation of coarsening, possibly on the cell walls, in an ensemble of droplets. Narrow path-length is a prerequisite for establishing transparency quickly in high droplet density media.

These experiments began while submitting a new proposal to continue ground-based testing. Postponement of flight preparation is a consequence of the inadequacy of hardware suggested for flight. The Bubble Drop and Particle Unit was not appropriate because of its inefficiencies regarding thermal fluctuations. Unless approximately one millikelvin thermal control is available, spurious nucleation and droplet dissolution will disrupt distribution self-similarity, critical to late-stage coarsening theory.

## II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/89    EXPIRATION: 12/93

PROJECT IDENTIFICATION: 963-24-08-01

NASA CONTRACT No.:    In-house

RESPONSIBLE CENTER: MSFC

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### *Microscale Hydrodynamics Near Moving Contact Lines*

PRINCIPAL INVESTIGATOR: Prof. Stephen Garoff

Carnegie Mellon University

CO-INVESTIGATORS:

M. Weislogel

NASA Lewis Research Center (LeRC)

#### TASK OBJECTIVE:

The objective of this study is to characterize the relationship between macroscopically observed dynamic contact angles and the microscopic fluid physics occurring near the contact line.

#### TASK DESCRIPTION:

This task consists primarily of low-gravity experimental studies on moving contact lines. The experiments will simultaneously measure flow fields and the fluid/fluid interface very near the moving contact line using video microscopy and particle image velocimetry. The experiments will be conducted in low gravity since it is in low gravity that interfacial phenomena dictate the fluid/fluid interface shape and location.

#### TASK SIGNIFICANCE:

The essential points of the investigation are:

1. Are the viscous stresses near a moving contact line appropriately described by present phenomenological models?
2. Can predictive models for spreading be developed for complex systems (rough surfaces, non-Newtonian fluids, etc.) if the flow fields and interface shapes near a contact line are known?
3. What is the nature of the unique fluid dynamical processes near a moving contact line? How do these processes depend on the properties of the fluids and the solid surface involved? How do these processes determine the true dynamic contact angle?

#### PROGRESS DURING FY 1994:

Our efforts this year focused on two areas: (1) working with the engineering team to refine science requirements for the flight experiment, consult on hardware design, prepare the Science Requirement Document, and present the Science Concept Review and (2) ground-based research on the moving contact line problem.

Definition of science requirements and discussions on hardware design for the abbreviated ("fasttrack") flight experiment were a major fraction of the total effort during the past contract year. Cell dimensions were set by calculations of static interface shapes, critical bond numbers, and recirculation flows. The requirement on thermal gradients in the experiment was chosen through a comparison of residual Marangoni flow and the driven flow under investigation. New UV cleaning chambers were built and tests were run on the effectiveness and resiliency of the UV cleaning process. In several meetings and correspondence, details of the cleaning techniques were transmitted from the science team at CMU to the engineering team at NASA Lewis Research Center. The science objectives of the flight experiment were refined and a minimal flight test matrix was established. Requirements were established for optical design, materials selection and characterization, data acquisition, temperature and pressure control, as well as acceleration levels and measurement. All requirements were recorded in drafts of the Science Requirement Document. The Science Concept Review occurred on May 4, 1994. This review presented the scientific motivation and experimental plans only for the "fasttrack" flight experiment.

Our ground-based research on moving contact lines produced important results both on the hydrodynamics in the intermediate region and on the inner scale physics:

We have measured the dynamic interface shape over long distances ( $\approx 2\text{mm}$ ). We found that the parameter  $w_0$  determined by fitting the dynamic data near the contact line to the composite model is the contact angle for the interface shape far from the contact line fit to a static interface shape. This result confirms a fundamental hypothesis of the model of the microscale hydrodynamics near a moving contact line. However, within the accuracy of our experiments, the interface shape at  $Ca$ 's as low as 0.01 is measurably different from the static shape even at distances of 1.2mm from the contact line. This result raises questions about the accuracy of measurements of dynamic interfaces made by other techniques and reported in the literature.

By measuring the interface shape at increasing  $Ca$ , we have determined the limits of the presently available models for the hydrodynamics near moving contact lines. We have made the first measurements of dynamic interface shapes in depression. We find that the present models are adequate for  $Ca < 0.1$ . Above this  $Ca$ , the model predicts too large a curvature at small distances from the contact line. The region where the model is inadequate expands as  $Ca$  increases. The breakdown is either due to an insufficient number of terms in the  $Ca$  expansion describing the intermediate region or to the inner scale hydrodynamics affecting the interface shape at unexpectedly large distances. The model of the intermediate region can be extended to higher  $Ca$ . It then provides an additional parameter describing the inner scale physics. However, our measurements show that this expansion cannot be used in terrestrial measurements because of contamination from geometry dependent terms which cannot be expressed analytically. This result proves that microgravity measurements, which increase the outer length scale, will provide a unique opportunity to measure an additional parameter with which to characterize inner scale physics.

Our materials studies show that the inner scale hydrodynamics of polymer melts is sensitive to local polymer/surface interactions. In a low  $Ca$  regime, the spreading of polydimethylsiloxanes (PDMS) across a 7740 Pyrex surface is strongly affected by the end termination chemistry of the polymer: hydroxyl terminated PDMS exhibiting a strong velocity dependence and methyl terminated PDMS exhibiting a weak dependence. Further, the hydroxyl terminated spreading varies with polymer viscosity in this regime. At higher  $Ca$ , the behavior of PDMS with either termination is similar. We are presently investigating scaling relations in this high  $Ca$  regime. We have also found that the spreading behavior of the hydroxyl terminated PDMS is strongly dependent on the level of absorbed water on the Pyrex surface.

Initial measurements of the velocity field near the moving contact line have been made. The PDMS is seeded with particles of  $Al_2(OH)_3$  coated with oleic acid.

The particles are in 1-10  $\mu\text{m}$  in diameter. Particle trajectories and velocity fields are measured and compared to the fields which are predicted by the modulated wedge approximation central to the model of the intermediate region which we use to fit the interface shape. At low  $Ca$ , the model correctly predicts the flow field down to distances on the order of 50  $\mu\text{m}$  from the contact line. These studies will continue probing closer to the contact line and higher  $Ca$  flows.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 3

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-25-0A-81

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Willson, K.R. and Garoff, S. Effect of chain termination chemistry and molecular weight on dynamic wetting of polymer liquids. to be published in Colloids and Polymers. (August 1994).

### Proceedings

Garoff, S., Chen, Q., Rame, E., and Willson, K.R. "Microscale hydrodynamics near moving contact lines." Proceedings of the 2nd NASA Microgravity Fluids Conference, Cleveland, OH, June, 1994.

### Presentations

Chen, Q., and Garoff, S. "Dynamic contact angles and interface shapes at high capillary number." American Physical Society Meeting, Pittsburgh, PA, March, 1994.

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Willson, K.R. and Garoff, S. "Dominant factors controlling dynamic wetting of PDMS fluids." 68th ACS Colloid and Surface Science Symposium, Stanford, CA, June 1994.



*Plasma Dust Crystallization*

PRINCIPAL INVESTIGATOR: Prof. John A. Goree

University of Iowa

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this research is to demonstrate that charged particulates in a plasma environment form an ordered structure.

**TASK DESCRIPTION:**

The investigator will design and test apparatus for radio-frequency (RF) plasma generation / particulate confinement, and analyze the structure of suspended particulates. This will include working with German scientists in designing microgravity experiments.

**TASK SIGNIFICANCE:**

This investigation begins a new genre of microgravity research. It will provide: (1) a macroscopic model of how atoms arrange themselves in a crystal or liquid, (2) a much-needed test of dusty plasma theories used in space physics and astronomy, and (3) an understanding of particulate contamination during plasma processing steps of microchip manufacturing.

**PROGRESS DURING FY 1994:**

This project represents an entirely new area of scientific research. When micron-size particulates are inserted into a plasma, they become charged and can arrange themselves in an ordered structure, ranging from a liquid to a crystalline lattice. This structure is suspended in the vacuum, and it is visible to the unaided eye. This is of interest for basic science, since it serves as a model system for studying crystalline structures and phase transitions to more disordered states. It is also of interest for industry, since we use apparatus identical to the tools used to etch silicon wafers in semiconductor manufacturing; particulates are of great importance because they can contaminate the silicon wafer. Gravity is a significant force acting on these particulates.

Experimental progress:

1. We have submitted and published in Physical Review Letters a paper describing for the first time an observation of a crystalline lattice of charged grains suspended in a plasma. This paper has generated considerable interest, and news articles about it have been written in *Nature*, *Physics World*, and *Science News*.
2. A new laboratory test stand has been assembled, and we have now used it to prepare more extensive data. We now have images of our particulate cloud made by video cameras pointing at sheets of laser light that cut through the cloud in vertical and horizontal cross sections. These experiments are yielding very clear images of ordered states ranging from liquid to hexatic regimes. We have analyzed these data using pair and bond-angle correlation methods developed originally by experimenters in colloidal suspensions. These experiments serve the dual role of providing exciting experimental results in their own right, helping to define this new area of research, while also serving as an early model for developing flight hardware. It is increasingly clear from the data that gravity crushes our particulate cloud into an essentially 2-D ordered structure, and that microgravity conditions will be required to achieve a fully 3-D ordered state.
3. In cooperation with our DARA-funded German collaborators, we are now discussing apparatus configurations for

a future Get Away Special flight experiment. In this international scientific collaboration, the PI is the senior experimenter.

### Theory progress:

We have begun a second theoretical effort, having already completed a first during FY93. We will model the dynamics of the particulates in our plasma. This effort will continue over the next year, with a visiting scientist from Norway doing much of the work. It will help in understanding our experiments and in quantifying the role of gravity in them.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 963-24-08-02

NASA CONTRACT NO.: NAG8-292

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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#### **Presentations**

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Goree, J. "Dusty plasma experiments and theory." UCSD Plasma Physics Seminar, June, 1994.

Goree, J. "Dusty plasma experiments and theory." Invited talk at the Workshop on Non-Neutral Plasma, July, 1994.

Praburam, G., and Goree, J. "Observations of particle layers levitated in an rf sputtering plasma." Gaseous Electronics Conference, Montreal, Canada, October, 1993.

Praburam, G., and Goree, J. "Observations of particle layers levitated in an rf sputtering plasma." American Physical Society Meeting, Division of Plasma Physics, November, 1993.

### *Evaporation from a Meniscus within a Capillary Tube in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Kevin P. Hallinan

University of Dayton

CO-INVESTIGATORS:

Dr. J. Ervin

University of Dayton

#### **TASK OBJECTIVE:**

The primary objective of this project is to investigate the thermocapillary effect on the interfacial instability of an evaporating extended meniscus and to corroborate the disjoining pressure gradient theory for describing the thin film fluid flow.

#### **TASK DESCRIPTION:**

In order to effectively study the thin film region of a capillary interface, experiments will be conducted in a large evaporation/condensation loop in a low-gravity environment. The experiments will include simultaneous particle image velocimetry (PIV), interferometry, and wall temperature measurement in order to obtain the fluid velocities, the interface shape, and the wall temperature in the thin film region. Thermocapillary effects will be quantified by the measured wall temperature and the fluid velocity profile in the thin film region.

#### **TASK SIGNIFICANCE:**

1. The study of the thermocapillary effect on the interfacial instability of an evaporating extended meniscus may help explain the failure of Capillary Pump Loops in a microgravity environment. The knowledge obtained from this study could result in radical new heat exchanger technologies that will benefit both space exploration and terrestrial applications.
2. This study can help strengthen support for the use of disjoining pressure gradient theory to describe fluid flow in thin films. Such an understanding can complete the theory of thin-film physics which is relevant to many important technologies from the thin-film coating to materials processings.

#### **PROGRESS DURING FY 1994:**

In FY94, numerical simulation of the thermo-fluid physics in the near contact line region was conducted to better understand the competing effects of thermocapillary stresses which oppose the liquid motion into this region and the disjoining pressure gradient which acts as the driver for the flow in this region. It was shown that a temperature difference of as little as 0.001K between the wall and the vapor could significantly affect the thin film flow field and marginally affect the total thin film heat transport and shape. The macroscopic influences of the thermocapillary stresses at near contact line were also observed experimentally by applying heat input to an evaporating liquid in a small-diameter, vertical capillary tubes. As the heat input to the meniscus was increased, the meniscus receded from the static equilibrium position which, by scaling arguments, is attributable to near contact line thermocapillary stresses. In addition, progress has been made in the continuing development of the Coherent Forward Scattering Particle Velocimetry technique which ultimately will be used to measure the velocity field in the evaporating thin liquid film near the contact line. An analytical analysis has identified the lengthscales near the contact line where the thermocapillary stresses can be most severe. Knowledge of the salient lengthscales has provided the guidance for selecting the optics and light source to be employed to extract information near the contact line. Polystyrene spherical seeding particles, 0.1 microns in diameter, will be the fluid tracers used to measure the near contact line velocity field. A Nikon Labophot microscope with Koehler illumination and having a 60X, 0.85 N.A. microscopic objective lens has been proven capable of providing reasonable diffraction images of particles in and out of focus inside of an evaporating liquid film.

## II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 3  
MS Students: 0  
PhD Students: 3

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-25-0A-21

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

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#### Presentations

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*Geophysical Fluid Flow Cell*

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PRINCIPAL INVESTIGATOR: Dr. John E. Hart

University of Colorado, Boulder

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## CO-INVESTIGATORS:

F. Leslie

NASA Marshall Space Flight Center (MSFC)

T. Miller

NASA Marshall Space Flight Center (MSFC)

J. Toomre

University of Colorado, Boulder

D. Ohlsen

University of Colorado, Boulder

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**TASK OBJECTIVE:**

The Geophysical Fluid Flow Cell experiment (GFFC) takes advantage of the unique environment of the microgravity laboratory, which permits forces that would otherwise be swamped by normal terrestrial gravity to become dominant. The GFFC uses electrostatic forces to warp gravity into a radial vector field, centrally directed towards the center of the cell. This allows us to perform visualizations of thermal convection in a spherical shell of liquid subject to imposed differential heating and basic rotation, where the active buoyancy forces are radially directed, as in planetary atmospheres and stars. The objective of the experiments is to categorize the types of convective patterns that can occur in highly nonlinear and turbulent flows with varying amounts of basic rotation and thermal stress.

**TASK DESCRIPTION:**

1. A significant modification to the original GFFC experiment that flew previously on Spacelab-III is the addition of real-time video visualizations of the fluid turbulence that can be downlinked from the shuttle to scientists on the ground. This permits interactive experiments that can identify and focus on important flow regimes. Design, construction and verification of space qualified hardware to effect this video downlink is a major hardware task.
2. In preparation for flight, theoretical, computational, and terrestrial laboratory simulations of rotating convection are required in order to suggest particular experiments that can critically advance our basic understanding of such phenomena, as well as make contributions to the science of geophysical and astrophysical fluid dynamics. Theoretical and computational models typically involve assumptions that will be checked in the spacelab flights, and terrestrial experiments are planar in geometry, allowing comparison of fundamental differences between "flat" and "spherical" convection.

**TASK SIGNIFICANCE:**

The spherical configuration and radial gravity of the Geophysical Fluid Flow Cell is significant because large-scale motions of the atmospheres of planets and stars are constrained by the inherent spherical geometry of these bodies. Furthermore the flows on them are constrained by rotation, under the action of same Coriolis forces that shape Earth's weather, and by buoyancy forces which result in hot fluid rising in a radial direction. It is impossible to study such motions in the terrestrial laboratory because gravity is uniformly directed. The GFFC experiments will provide basic laboratory data that can be applied to problems of cloud patterns on the giant planets, differential rotation on the Sun, and motions in Earth's core, mantle and atmosphere.

**PROGRESS DURING FY 1994:**

1. The GFFC Video Acquisition Module (VAM) and associated documentation were completed and shipped to MSFC for integration with the GFFC. Tests there indicate the VAM is functioning properly.
2. The computational code of Gary Glatzmaier, previously used with the SL3 GFFC results, was upgraded. Now more resolution can be used, and it will be possible to computationally simulate most of the anticipated GFFC USML-2 runs. Research Associate Dr. Dan Ohlsen will begin running cases to be studied on USML-2 in early FY95.

3. A major laboratory experiment on turbulent rotating convection was constructed. It permits in situ thermal measurements and remote measurement of velocity fields of rotating convection using particle image velocimetry (PIV). Initial results show strong cyclonic vortices (tornado turbulence) which are associated with a strong unstable interior stratification (compare with non-rotating convection where the interior stratification is neutral).

4. Experiments and computational simulation of b-convection (rotating convection constrained to lie near the equator where gravity is perpendicular to rotation) were completed. The computational simulations show strong jets (like Jupiter), but the experiment, which run at higher Prandtl number, do not. We intend to look more closely at this problem in FY95.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/88 EXPIRATION: 3/94

PROJECT IDENTIFICATION: 963-24-08-04

NASA CONTRACT NO. NAS6-31958

RESPONSIBLE CENTER: MSFC

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*Interfacial Phenomena in Multilayered Fluid Systems*

PRINCIPAL INVESTIGATOR: Prof. Jean N. Koster

University of Colorado, Boulder

CO-INVESTIGATORS:

Prof. S. Biringen

University of Colorado, Boulder

**TASK OBJECTIVE:**

The main objective of the research is to design a three-liquid-layer flight experiment that will study the interaction of two interfacial tension forces of different magnitude, and their effects on thermocapillary fluid flow induced in adjacent liquid layers. The thermocapillary flow results from temperature gradients parallel to the fluid interfaces. In addition, the mechanical coupling between the immiscible layers and the suppression of convective flow will be investigated. The conditions for the existence for oscillatory flow, and the effects of g-jitter may also be studied.

**TASK DESCRIPTION:**

The general approach is to conduct ground-based normal-gravity testing and develop theoretical models of the combined buoyant and thermocapillary convection phenomena. The instrumentation and diagnostics are centered around the physics of interest; namely, flow fields, temperature fields, and interfacial shapes. The theories and numerical models developed and verified with the 1-g data was used to predict the results of the flight experiment on the IML-2 mission.

**TASK SIGNIFICANCE:**

The results are expected to significantly advance our knowledge in the area of surface-tension-driven convection in multilayered fluid systems. The scientific results will find applications related to encapsulated float zone processing. Float zone processes are a technique in which space processing of crystals can be done while minimizing imperfections.

**PROGRESS DURING FY 1994:**

There were many activities on the flight project side. Activities were concentrated on preparing for and in support of the flight experiment in July 1994. These activities included participating in ground testing of the engineering/flight models in Europe; familiarizing the crew with science background of the experiment; and attending various mission simulations at MSFC. During the course of the year, the PI expressed concerns about maintaining liquid-liquid interfaces during curtain retraction, having sufficiently small particles in the liquids to achieve the required velocity field resolution, and having equal pressures in each of the three fluid layers so that the interfaces remained flat. During the actual mission in July, flat interfaces were, unfortunately, lost when the first curtain withdrawal was nearly half completed. The final fluid configuration (when both curtains were retracted) was the undesirable shape of a fluorinert ball surrounded by silicone oil. Although some tests were subsequently done with this configuration, it is felt that little scientific data resulted from the experiment.

Ground-based research continued in the PI's labs in Boulder during the year. Research was conducted in the following areas: natural convection in multilayer systems; combined thermocapillary and natural convection in multilayer systems; Rayleigh Benard convection in multilayer systems; and assessing the effect of g-jitter on fluid interfaces. The work for the next year is currently being replanned in light of the flight experiment results. One of the priorities of the work to be done will be to better understand how to establish such interfaces in microgravity.

## II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 4

TASK INITIATION: 11/89 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-24-00-05

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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*Extensional Rheology of Non-Newtonian Materials*

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PRINCIPAL INVESTIGATOR: Prof. Gareth H. McKinley

Harvard University

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this work is to determine the extensional viscosity in uniaxial stretching flow of fluid systems such as dilute polymer solutions, emulsions, and suspensions. Also, to generate a simple homogeneous shear free flow in the material, and to measure the stress response of the material from an imposed rate of deformation (constant deformation rate experiments).

**TASK DESCRIPTION:**

This effort will require an initial ground-based experimental design, and ultimate payload implementation, of a novel experimental apparatus to measure accurately the rheological response of non-Newtonian fluids under shear-free conditions that are characteristic of those experienced in the containerless processing of materials.

The proposed instrument generates a homogeneous uniaxial elongation through an exponential stretching of the test sample, and the spatial uniformity of the deformation rate experienced by the fluid is verified by digital particle image velocimetry. Direct measurements of the tensile force exerted by the material then allow calculation of the extensional viscosity of the fluid.

The design of the apparatus utilizes several of the existing or planned fluid diagnostic modules being considered by NASA, and in addition, provides a completely new fluid-science flight capability, which can be used repeatedly to support multi-user rheological measurements for a wide range of non-Newtonian fluids.

**TASK SIGNIFICANCE:**

The extensional viscosity is a fundamental physical property of all non-Newtonian materials, and cannot be determined from simple viscometric shear flow experiments. Constitutive equations for viscoelastic fluids such as dilute polymer solutions predict large changes in the extensional viscosity as the elongation rate is increased; however, the validity of these theories cannot be confirmed due to the lack of experimental data obtained in extensional flows. To date, quantitative rheological measurements in shear-free flows have only been possible for highly elastic or "stiff" materials such as polymer melts which can easily be elongated without sagging under a gravitational body force. By performing similar experiments in an extended microgravity environment it will be possible for the first time to obtain accurate measurements of the extensional viscosity for more 'mobile' fluids such as polymer solutions, suspensions and liquid crystalline materials. This rheological data will allow designers of both space- and ground-based material processes to use improved constitutive models in numerical simulations of complex two- and three-dimensional fluid flows.

In a simple stretching flow, fluids such as water or syrup exhibit a resistance to stretching that is exactly three times the value of their Newtonian viscosity. This resistance is usually termed the "extensional viscosity of the material." However, for fluids containing long macromolecules (e.g., synthetic polymers, liquid crystals, or DNA) is predicted that the extensional viscosity can be anywhere from 100 to 10000 times greater than the viscosity of the fluid. In the near future, NASA plans to develop "containerless processing" operations under microgravity and a detailed understanding of extensional properties in fluids will be absolutely critical -- since, in the absence of container walls, the only way to mix, pour, and shape fluids will be through pure stretching motions. Knowledge of such nonlinear material functions is fundamental to the development and verification of relatively cheap ground-based computational modeling techniques which can be used effectively in the "a priori" design of advanced

microgravity material processes thereby alleviating the need for costly in-flight pilot experiments. Finally, in addition to their significance in many industrial processes such as fiber-spinning and film coating operations, stretching flows of non-Newtonian fluids are of fundamental scientific importance to a number of complex fluid dynamics phenomena, including the stability and breakup of jets, enhanced oil recovery and turbulent drag reduction for advanced aircraft, boats, and submarines.

### PROGRESS DURING FY 1994:

The second draft of the Science Requirements Document has been submitted and the Science Concept Review was successfully completed on May 6, 1994. A Boger (polymer solution) Fluid Column deployment breadboard and an exponentially grooved wheel breadboard for the neutral density tank were completed. A dry wheel breadboard has also been constructed, and tests are being planned. The particle image velocimetry apparatus has been built and is being tested at LeRC.

Future plans include further development and construction of the following:

1. The drive mechanism breadboard testing - Inertial motion of neutral density fluid: viscous diffusion of Boger fluid
2. Birefringence tests
3. Optical testing: DPIV software integration, particle and laser selection and radial measurement
4. Reducing diameter device (RDD) breadboard testing
5. Load cell and microbalance testing (.01 to 30 grams range required)
6. Sridhar Apparatus testing - Fixed plates then RDD with electronic balance
7. Adiabatic heating - Theoretical estimation
8. Fluid choice - rheological analysis of varying concentrations of polyisobutylene (monodisperse)
9. Lubrication - Analysis of slip on wheels in plateau tank
10. Surface tension - Determine correlation of dynamic surface tension to extensional rheology experiment.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-25-0A-36

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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*Pool Boiling Experiment*

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PRINCIPAL INVESTIGATOR: Prof. Herman Merte, Jr.

University of Michigan

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The program described here seeks to improve the understanding of the fundamental mechanisms that constitute nucleate pool boiling. The vehicle for accomplishing this is an investigation, including experiments to be conducted in microgravity and coupled with appropriate analyses, of the heat transfer and vapor bubble dynamics associated with nucleation, bubble growth or collapse, and subsequent motion. Certain effects that can be neglected at normal Earth gravity, such as surface tension and vapor momentum, can become quite significant in microgravity. Momentum imparted to the liquid by the vapor bubble during growth tends to draw the vapor bubble away from the surface, depending on the rate of growth, which in turn is governed by the temperature distribution in the liquid. Thermophoretic forces, arising from the variation of the liquid-vapor surface tension with temperature, on the other hand, tend to move the vapor bubble toward the region of higher temperature. The bubble motion will be governed by which of these two effects prevail.

The elements of nucleate boiling, for which research conducted under microgravity would advance the basic understanding, are:

1. Nucleation or onset of boiling. Indications are that both heater surface temperature and temperature distribution in the liquid are necessary to describe nucleation.
2. The dynamic growth of a vapor bubble in the vicinity of the heater surface. This includes the shape as well as motion of the liquid-vapor interface as growth is taking place. These are influenced by the liquid temperature distribution at the initiation of growth.
3. The subsequent behavior of the vapor bubble. This includes the motion, whether departure takes place or not, and the associated heat transfer.

**TASK DESCRIPTION:**

In the proposed experiment, a pool of liquid—initially at a precisely defined pressure and temperature—will be subjected to a step-imposed heat flux from a semitransparent thin-film heater forming part of one wall of the container, such that boiling is initiated and maintained for a defined period of time at a constant pressure level. Transient measurements of the heater surface and fluids temperatures near the surface will be made, noting especially the conditions of the boiling process in two simultaneous views, from beneath the heating surface and from the side. The conduct of the experiment and the data acquisition will be completely automated and self-contained. For the initial flight, a total of nine tests are proposed, with three levels of heat flux and three levels of subcooling.

**TASK SIGNIFICANCE:**

The outcome of the experiment is expected to include the following:

1. Observation of the liquid-vapor behavior, including bubble growth and motion as functions of heat flux, initial subcooling and time, and correlation with observed heater surface temperature variation.
  2. Use of initial liquid temperature distribution at nucleation to compute vapor bubble growth rate for comparison with observation.
  3. Measurement of delay time to nucleation for correlation with nucleation theory.
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Anyone who has ever boiled water on a stove is familiar with nucleate pool boiling. Even though it is an everyday event, scientists do not understand precisely how it works, because Earth's gravity influences how bubbles form and grow in boiling liquids.

NASA is interested in results from this experiment, because boiling liquids generates bubbles which are very efficient at transferring large amounts of heat. Finding new ways to dissipate heat from the space shuttle or future manned space platforms will be vital to the success of long term missions.

There are potential benefits closer to home as well, including more effective air conditioning and refrigeration systems and improvements in power plants that could reduce the cost of generating electricity.

### PROGRESS DURING FY 1994:

The prototype hardware for the Pool Boiling Experiment was flown aboard the SL-J mission on September 12–20, 1992. Performance of the hardware was "near perfect." The data clearly reveal that pool boiling in reduced gravity ( $10^{-3}g$ ) is a transient process and not a steady periodic one. At the higher-heat flux tests ( $8 \text{ W/cm}^2$ ), the temperature, as well as the vapor content continued to increase. Tests conducted at the lower-heat flux levels resulted in a rapid spreading of the vapor across the heater as compared to the high-heat flux levels. In low gravity, the vapor bubbles adhered to the heater surface and were 1 cm to 5 cm in diameter. In normal gravity, the vapor bubbles lift off the heater surface due to buoyancy and are approximately 1.5 mm in diameter.

The flight hardware was flown on STS-57 mission in June 1993. Eight of nine test points were successful. The Pool Boiling Experiment was flown again on the STS-60 mission, in February 1994. All nine test points were successful.

The principal investigator submitted the final report on the results of the STS-47 mission to NASA-Lewis in July 1994. A combined final report of the STS-57 and STS-60 missions should be completed by January 1995.

The results from these three flights can be summarized as follows:

In microgravity the absence of buoyancy causes large bubbles to form and often remain on the heater surface as surface tension plays a dominant role. Some dryout and rewetting was observed. In normal gravity ( $1g$ ) convection causes the bubbles to depart from the heater surface while they are quite small.

Correlation of dry out area to mean,  $h_c$ , and boiling,  $h_b$ , (wetted) heat transfer coefficients and mean heater surface temperature,  $T_w$ , for microgravity pool boiling tests is first of its kind.

Professor Merte successfully passed a Microgravity Hardware Reflight Reviews held on June 24, 1994 on his proposed related experiment, "Study of Pool Boiling in Microgravity-Rewetting Following Dryout," was approved by NASA based on favorable evaluations from peer review panel. Two flights are planned using the same pool boiling hardware with a few modest changes. The first mission should occur in the spring or summer of 1995.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

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TASK INITIATION: 2/90 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 963-24-0B-10

RESPONSIBLE CENTER: LeRC

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*Surface Tension-Driven Convection Experiment (STDCE-1, STDCE-2)*

PRINCIPAL INVESTIGATOR: Prof. Simon Ostrach

Case Western Reserve University

CO-INVESTIGATORS:

Prof. Y. Kamotani

Case Western Reserve University

**TASK OBJECTIVE:**

The objective of this research is to further the understanding of the physical mechanisms associated with non-oscillatory (STDCE-1) and oscillatory (STDCE-2) thermocapillary flow by (a) developing an accurate description of the physical mechanisms, (b) developing an accurate numerical model, and (c) obtaining ground-based and flight experimental data to verify the physical mechanisms and the numerical model. The thermocapillary flows result from the fluid motions generated by the surface-tractive force that is caused by surface-tension variation due to the temperature gradient along the free surface.

**TASK DESCRIPTION:**

STDCE-1: The basis of Surface-Tension-Driven Convection Experiment 1 (STDCE-1) flight experiment is a copper test cell 10 cm in diameter and 5 cm deep, filled with silicone oil, able to provide both flat and curved free surfaces in a microgravity environment. The outer wall of the test cell is water cooled. The silicone oil can be centrally heated either externally by a carbon dioxide laser (constant heat flux, CF) or internally by an immersion heater (constant temperature, CT). The cross section is illuminated by a 1-mm-thick sheet of light, which scatters from small aluminum oxide particles mixed into the oil, allowing observation and measurement, using a particle-tracking technique, of the axisymmetric flow velocity. An infrared imager is used to measure surface temperature, and thermistors are used to measure fluid and wall temperature. The velocity and temperature measurements are compared with the numerical predictions.

STDCE-2: The center of Surface-Tension-Driven Convection Experiment 2 (STDCE-2) is an interchangeable module containing a test cell and fluid reservoir. Six modules containing copper test cells of 1.2, 2.0 and 3.0 cm diameter, each with the depth equal to the radius, will be filled with 2 centistoke silicone oil, to provide both flat and curved free surfaces in a microgravity environment. In three of the modules, one of each size, the fluid will be heated by a carbon dioxide laser, imposing a Gaussian heat flux on the free surface, and in the remaining three the fluid will be heated internally by an axially located heater which is ten percent of the chamber diameter. The outer walls of the test chambers will be cooled. This modular approach was taken to accommodate the large test matrix.

During the experiment, the surface temperature—which is the driving force in the flow—is measured non-intrusively by an infrared imager. The free-surface deformation, felt to play a critical role in the oscillation phenomenon, is measured quantitatively using a Ronchi deflectometer. The flow field is observed by illuminating the entire test chamber volume with laser light which is scattered from 20 micron aluminum oxide particles mixed in the fluid, allowing for three-dimensional qualitative visualization. Thermistors are used to measure bulk fluid, wall and heater temperatures.

At the start of each test the heater power will be slowly increased until the flow transitions from steady and axisymmetric to periodic and three dimensional. This will be performed for 43 combinations of test chamber size, heating mode and free surface shape. The temperature difference at the transition point will be used to calculate non-dimensional parameters which are used to characterize the onset of oscillations. The flow field, surface and bulk temperature distributions, and the free surface deformations will be correlated to support the proposed physical mechanism for the oscillatory phenomenon.

**TASK SIGNIFICANCE:**

The Surface Tension Driven Convection Experiment is designed to study the nature and extent of steady and oscillatory thermocapillary flows using state of the art diagnostics to measure and characterize these flows over a wide range of parameters. Valuable data, which can only be gained from low gravity-based experimentation, will be obtained resulting in an understanding of the fundamental physical mechanisms and improved implementation of related industrial processes such as life support systems, containerless processing of materials, crystal growth, propellant storage management, and bio-fluids engineering both in space and on earth.

**PROGRESS DURING FY 1994:****STDCE-1**

STDCE-1 was flown on USML-1 in June, 1992. 38 tests were completed returning over 12 1/2 hours of data. To date approximately 75% of the data has been analyzed and compared to the numerical model. The comparisons show good agreement. The analysis of the data has been broken into sections and numerous presentations of those sections of the reduced flight data have been made. These include flow data from the 1 hr CT and CF tests, temperature data from the 1 hr tests, flow data from the shorter CT flat surface tests, and flow data from the shorter CF curved surface tests. All of the data is presently being compiled by a graduate student for the final contract report. In addition, no flow oscillations were observed in any of the tests corroborating the Principal Investigator's theory that the Marangoni number alone is not sufficient to indicate the onset of oscillatory flow.

**STDCE-2****Flight Experiment:**

STDCE-2 is scheduled to fly on USML-2 in September of 1995. In preparation, the PI and Co-I participated in two USML-2 Investigator Working Group meetings at the Marshall Space Flight Center in addition to numerous meetings and reviews at the Lewis Research Center.

**Ground-based work:****1. Analysis**

A scaling analysis of axisymmetric thermocapillary flows was conducted. Various important velocity and length scales were determined. The scaling laws were shown to agree well with the results of a numerical analysis. Based on the scaling analysis a surface deformation parameter (S-parameter) was derived for each CT and CF heating mode. Both Marangoni number and the S-parameter must be larger than certain values in order to obtain oscillatory thermocapillary flow. The work has been written up as a Ph.D. thesis.

**2. Experimental Work**

The deformation of the free surface has been measured in oscillatory thermocapillary flow. The free surface motion was measured by observing the surface in one radial cross-section through a micro-video system. An organized free surface motion was found. The frequency of the free surface oscillations was the same as that of the temperature oscillations detected by a thermocouple probe in the fluid. Both oscillation patterns were closely related.

An optical system is being set up to measure the free surface motion over the entire free surface instead of on section. This optical measurement will provide us with information regarding a wave-like motion of the free surface. The information is important in establishing a coupling among the surface motion and the rotating temperature oscillation pattern.

An experiment on oscillatory thermocapillary flow in cylindrical containers is being performed. The objective is to confirm the S-parameters obtained by analysis. In order to do so the effect of buoyancy on oscillatory thermocapillary flow must be understood. The buoyancy effect is being investigated numerically and experimentally.

## II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	3
PhD Students:	3	PhD Degrees:	1

TASK INITIATION: 4/91 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 963-25-0D-09

RESPONSIBLE CENTER: LeRC

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### *Modeling and New Experiment Definition for the VIBES*

PRINCIPAL INVESTIGATOR: Prof. Robert L. Sani

University of Colorado, Boulder

#### CO-INVESTIGATORS:

Dr. H. Azuma	Japanese National Aerospace Laboratory (NAL)
Dr. T. Doi	NSDA — Japanese Space Agency
Dr. S. Kamei	Mitsubishi Research Institute
Dr. M. Ohnishi	Japanese National Aerospace Laboratory (NAL)
Dr. T. Kida	Japanese National Aerospace Laboratory (NAL)
Dr. K. Yamamoto	Japanese National Aerospace Laboratory (NAL)

#### TASK OBJECTIVE:

The Vibration Isolation Box Experiment System (VIBES) is an IML-2 flight experiment being designed by the Japanese National Aerospace Laboratory. Its primary goal is to evaluate the performance of a vibration isolation device in conjunction with typical fluids experiments. The IML-2 flight experiment will contain two experimental units: the Convection Diffusion Unit (CDU) and the Thermal-Driven Flow Unit. The purposes of the CDU experiment (the one of interest herein) are to observe natural convection and diffusive transport in a micro-g environment and to observe the effect of g-jitter with and without the vibration isolation due to the vibration isolation box. The objective of this project is to provide numerical modeling for the CDU experiment for aiding in design refinements and evaluation of terrestrial benchmark experiments as well as post-flight evaluation of the experimental data.

#### TASK DESCRIPTION:

The numerical modeling will utilize a Galerkin finite element algorithm for the linear momentum, energy and species balance equations using the Boussinesq approximation. This project will make comparisons of two codes (PI's research code and a commercial code, FIDAP) in a transient, 3-D calculation to determine their efficiency and accuracy. Timing comparisons will also be made between FIDAP and the research code. The numerical experiments will include example cases with and without the test cell being subjected to g variation; both single and multiple frequency variations will be considered. The numerical experiment will also consist of simulating the g-environment (to be provided by the Japanese research team) both inside and outside the isolation box. Comparison of these results should allow a quantitative assessment of the isolation capability of the apparatus.

#### TASK SIGNIFICANCE:

The microgravity environment available for space experiments is not quiescent but is subjected to significant background vibrations generated by aerodynamic and machinery vibrations, crew motion, etc. Such g-jitter can be relatively random in orientation and can attain significant magnitudes. There is a growing list of observations and data analyses that demonstrate the existence of significant g-jitter episodes and their potential for having very deleterious effects on many proposed flight experiments. A potential solution to this problem in the micro-g environment is the use of vibration isolation for the experiments which require it. The assessment of such an apparatus is one of the main thrusts of the research proposed in this project.

#### PROGRESS DURING FY 1994:

During this reporting period, the following have been accomplished:

1. The development, testing and benchmarking of the semi-consistent mass finite element projection algorithm for 2D and 3D transient Boussinesq flow has continued.
  - a. The algorithm has been modified in order to include the option of utilizing a skew-symmetric form of the advection operator as a prelude to the implementation of a variable time step capability. This option required the

implementation of an iterative solution technique, bi-conjugate gradient stabilized, appropriate for unsymmetric algebraic systems. This has been done and the algorithm has been tested and benchmarked against known solutions existing in the literature as well as solutions generated via the commercial code FIDAP for a spin-up of a lid driven cavity.

b. The development of a variable time step algorithm with local time truncation error control is currently being pursued.

c. The inclusion of the capability to model additional fields, for example, concentration, is being addressed. Its eventual complementation will be an essential tool in the definition of potential new flight experiments focused on the behavior of coupled fluids in a g-jitter environment.

2. Optimization and parallelization studies are underway.

a. Since the last progress report Prof. Sani has further improved the optimization of the algorithm as well as initialized an investigation of the potential parallelization of the algorithm via domain decomposition.

3. Initial time-varying gravity Boussinesq flow simulation has been done.

a. The simulation reported in the previous progress report has been completed and the results are being compared with experimental observations made by a Japanese research team.

4. Ongoing communication with the Japanese research team for VIBES has been maintained.

a. The data from the VIBES flight (IML2) experiment, i.e., video, accelerometer data, etc., should be available in the next few months and post-flight analysis of the data will be initiated.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 963-24-05-14

RESPONSIBLE CENTER: LeRC

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*Studies in Electrohydrodynamics*

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PRINCIPAL INVESTIGATOR: Dr. Dudley A. Saville

Princeton University

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

This research is designed to strengthen and test the experimental foundations of the theory of electrohydrodynamics. Electrohydrodynamic forces can be used to manipulate fluids, especially fluid interfaces. As such, they offer a means of controlling fluid motion on very small length scales.

**TASK DESCRIPTION:**

Theoretical studies will center on adapting the existing leaky dielectric theory for the stability of a fluid cylinder to account for pinning the contact line at the upper and lower boundaries in a liquid bridge configuration.

Experimental studies will be carried out to (1) evaluate the influence of ionic surfactants on conductivity so as to enable us to control the time scale of the electrohydrodynamic fluid motion and (2) test the existing electrohydrodynamic theory for the stability of a cylinder subject to an axial field with isopycnic systems.

**TASK SIGNIFICANCE:**

Despite substantial efforts over the past two decades, the foundation of electrohydrodynamics is weak. Relatively few experiments have been done to test the leaky dielectric theory, the most promising model of behavior, and much of the work has been of limited scope because of the need to use isopycnic systems to avoid sedimentation and hydrostatic pressure effects. This restricts the range of fluid properties that can be studied and as a result there are many gaps in our knowledge. In addition to its scientific importance, there are a wide range of applications where electrohydrodynamic phenomena play important roles.

**PROGRESS DURING FY 1994:**

## Experimental work:

1. We were able to identify an ionic compound with which enables us to control the conductivity of low dielectric constant liquids. We studied the conductivity of castor oil containing various amounts of the solute and showed that the solution behaved as an ohmic conductor. The use of this system will enable use to control the time scale of the electrohydrodynamic motion.

2. The initial stages of the collapse of fluid cylinders was studied and we showed that the linearized theory was capable of representing the experimental results over the initial period. Unfortunately, the need to use very viscous fluids to control sedimentation limited the range of our results.

## Theoretical work:

We tested the existing theory for infinite cylinders against all the known asymptotic results and found that it was internally consistent. The next task is to adapt it to liquid bridges (pinned cylinders).

## II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 963-24-08-03

NASA CONTRACT NO.: NAG8-969

RESPONSIBLE CENTER: MSFC

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*Mechanics of Granular Materials*

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PRINCIPAL INVESTIGATOR: Dr. Stein Sture

University of Colorado, Boulder

CO-INVESTIGATORS:

N. Costes

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

The objective of this research is to examine the use of microgravity to gain a quantitative understanding of the mechanical behavior of cohesionless granular materials under very low confining pressures.

**TASK DESCRIPTION:**

Ground-based displacement-controlled triaxial experiments are conducted on a cohesionless granular material at the lowest effective confining pressures possible, that do not result in material instability, to assess constitutive properties, stability phenomena, and control parameters that will be applied to in-space experiments on 75 mm (diameter) and 150 mm (length) right cylindrical specimens. The ground-based tests on similar-sized specimens are conducted in the range 3.5-69 kPa, while the microgravity tests will be conducted at effective confinement levels in the range 0.05-1.30 kPa.

The displacement-controlled mode of loading confined specimens was chosen mainly to maintain overall specimen-apparatus stability while strain-softening resulting from continuous or discontinuous bifurcation and discontinuous deformation fields are allowed to take place. Optical and other noncontacting displacement-sensing techniques are used to measure specimen response during experimentation. Prescribed displacements are transmitted in terms of loading, unloading, and reloading histories, while volume change is measured in "drained" tests, and pore fluid pressure is measured in "undrained" isochoric tests. Confinement pressure is transmitted to the granular material assembly through a thin flexible latex membrane surrounding the specimen. A subangular and uniform Ottawa quartz sand constitutes the specimen.

Specimens tested both in space and on ground will be subjected to nondestructive and destructive (thin-slicing) testing to assess degrees of material uniformity and isotropy before and after experimentation. It appears that instability phenomenon associated with specimens of certain configurations result in curved internal surfaces of localized deformation and high rates of dilatancy, whose structure depends on bifurcation mode.

**TASK SIGNIFICANCE:**

Specifically, the purpose is to study the influence of particle interlocking and other fabric properties on the strength criterion near the effective stress space origin, i.e., can it be represented by a straight-line envelope passing through the origin or does it have a curved shape with shear (cohesion or interlocking) or tensile strength intercepts. The experiment will determine whether cohesionless granular materials under very low effective confining pressures/effective stresses tend to dilate or contract regardless of their initial state of compaction, and whether their mechanical behavior under relatively large displacement or quasi-static cyclic loading is according to conventional constitutive theory. In addition, bifurcation and material instability phenomena resulting in formation of shear bands, before and after peak strengths have been reached, will be studied. Based on terrestrial experiments and theory, it has been found that critical hardening, strain-softening behavior, and shear band orientation are dependent on confining stress.

### PROGRESS DURING FY 1994:

The progress in the MGM project during FY94 includes the following:

1. Design of the MGM apparatus;
2. Improved specimen preparation technique;
3. Improved optical-imaging system and measurement of specimen motion;
4. Improved analysis of specimen bifurcation instability conditions;
5. Improved constitutive analysis methods.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	2	PhD Degrees:	0

TASK INITIATION: 12/92    EXPIRATION: 9/97

PROJECT IDENTIFICATION: 963-24-08-05

NASA CONTRACT NO. NAS8-38779

RESPONSIBLE CENTER: MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Macari, E.J.P., Sture, S., and Runesson, K. Prediction of response of granular materials at low effective stress levels. *J. Geotechnical Engineering*, vol. 120, no. 7, 1252-1268 (1994).

Peric, D., Sture, S., and Runesson, K. Effect of pore fluid compressibility on localization in elastic-plastic solids. *Int. J. Solids and Structures*, vol. 32, no. 4, (1994).

#### Proceedings

Peric, D., Runesson, K., and Sture, S. "Effect of pore fluid compressibility on localization of elastic-plastic solids under undrained conditions." *Proceedings of the US National Congress for Applied Mechanics*, Seattle, WA (1994).

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*Thermocapillary Migration and Interactions of Bubbles and Drops*

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PRINCIPAL INVESTIGATOR: Prof. R. S. Subramanian

Clarkson University

Co-INVESTIGATORS:

Dr. R. Balasubramaniam

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objectives of the research are to experimentally measure the thermocapillary migration velocities and the shapes of single and interacting gas bubbles, and liquid drops in a continuous phase under the action of an applied temperature gradient. Comparisons between the observed velocities and shapes with those that are predicted from theory will be made.

**TASK DESCRIPTION:**

The general approach has been to conduct ground-based normal-gravity testing and to develop theoretical models of the thermocapillary migration phenomena. The instrumentation and diagnostics are centered on the physics of interest, namely, flow fields, temperature fields, and bubble/droplet velocities and shapes. The theories and numerical models developed and verified with the 1-g data will be used to design and predict the results of the flight experiment. Preliminary assessment of flight data will also begin within FY94.

**TASK SIGNIFICANCE:**

The results from these bubble migration experiments are not only expected to advance our knowledge in the area of surface tension driven motion, but are, in addition, relevant to several applications with respect to space processing of materials. Some examples of the latter include solidification, glass processing, and composite preparation. The physics studied in the experiments offer a method by which undesirable void formation in metals and composites can be avoided.

**PROGRESS DURING FY 1994:**

There were a lot of activities on the flight project side. Activities were concentrated on preparing for and in support of the flight experiment in July 1994. These activities included participating in ground testing of the engineering/flight models in Europe; in mission sequence testing of engineering models at KSC; providing test plans for the low-g aircraft flights that took place in July; familiarizing the crew as to the science background of the experiment; and attending various mission simulations at MSFC. During the course of the year, the PI expressed concerns about being able to inject single bubbles; about being able to extract bubbles; and about getting reliable PDI data. During the actual mission in July, the injection and extraction systems performed well, but little useful PDI data was obtained. The PI and his team are currently analyzing flight data results which will be reported on in the next period.

Ground based research continued in the PI's labs at Clarkson. Research was conducted in the following areas: migration of a pair of bubbles in a vertical temperature gradient; the study of drops held fixed under the simultaneous action of thermocapillary and gravitational effects; and the development of predictions of free convection due to transient gravitational fields.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

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TASK INITIATION: 1/90 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-25-0C-61

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Wei, H. and Subramanian, R.S. Interactions between two bubbles under isothermal conditions and in a downward temperature gradient. *Physics of Fluids A*, A6, 9, 1971-78 (1994).

#### Presentations

Forbes, Rashidnia, N. and Balasubramaniam, R. "Abel inversion of irregularly spaced discrete interferometric fringe data: A geometrical approach." Division of Fluids Dynamics Meeting, American Physical Society, Albuquerque, New Mexico, November, 1993.

Kondos, P.A. and Subramanian, R.S. "Buoyant flow driven by a time-dependent gravitational field in a two-dimensional cavity." AiChE Annual Meeting, San Francisco, November 1994.

Subramanian, R.S., and Kondos, P.A. "Gas bubble dissolution and growth in glass melts." invited review, American Ceramic Society Meeting, Indianapolis, Indiana, April 1994.



*Drop Dynamics Investigation*

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PRINCIPAL INVESTIGATOR: Prof. Taylor G. WangVanderbilt University

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## CO-INVESTIGATORS:

C.P. Lee

Vanderbilt University

A.V. Anilkumar

Vanderbilt University

A.B. Hmelo

Vanderbilt University

H.J. Hussein

Vanderbilt University

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**TASK OBJECTIVE:**

The objective of this program is to understand the behavior of free drops, primarily by studying them in a microgravity environment. The Drop Physics Module (DPM) operated in the Space Shuttle provides an opportunity to address outstanding fluid-dynamics issues of rotating and oscillating simple and compound drops. To maximize the return from this short on-orbit opportunity, ground-based experiments will be performed to verify concepts and experimental techniques, and modeling will be done to select the parameters for the DPM experiments.

This investigation will use a triple-axis acoustic positioning chamber to study the static behavior and dynamics of simple and compound drops as well as of liquid shells. Equilibrium shapes and the stability of rotating and nonrotating drops, their associated internal flow patterns, and the centering force associated with shape oscillatory dynamics of rotating compound drops—will be the principal scientific areas of interest.

**TASK DESCRIPTION:**

A variety of experiments will be performed in space. Compound drops and liquid shells will be formed to study their oscillation modes and the effectiveness of those modes in centering the core. The interaction between the acoustic field and the drops will be studied: the drops' static shape, the stability of distorted shapes, and the generation of any flows in the liquid. The dynamics and stability of rotating drops near the point of fission will be explored.

**TASK SIGNIFICANCE:**

This investigation uses the low gravity provided by the Space Transportation System, the working laboratory of Spacelab, and the Drop Physics Module hardware to study large drops. Studying drops from 1-3 cm. in diameter shows dynamic phenomena to time scales which can be observed by the experiment operator as well as captured on high-speed film.

**PROGRESS DURING FY 1994:**

Due to the late arrival of the DPM video data, the reduction and analysis of most of the USML-1 data was performed in FY 94; three papers have been prepared using this data and have been submitted for publication. Support to the DPM Project in the area of understanding tumble rotation progressed from flow visualization experiments at Vanderbilt to preparations for torque measurements on the flight system at KSC.

One of the primary objectives of USML-1 was to perform careful experiments on rotating drops. The DDM results from Spacelab-3 deviated from analytical and numerical predictions. The USML-1 results showed that flattening due to acoustic forces would cause experimental measurements to deviate from theoretical predictions that assume a spherical geometry; when the drops are spherical, there is no discrepancy. The results will be published in the *Journal of Fluid Mechanics* (Oct. 94).

Another set of USML-1 experiments studied the tendency of compound drops to become concentric under the influence of capillary oscillations. A compound drops is composed of two relatively immiscible fluids one totally

contained within the second fluid drop. Studies of both bubbles and liquid-liquid compound drops showed that the core slowly oscillated about the center and after several periods remained concentric. These results which can not be explained by existing inviscid models were published in the Journal of Colloid and Interface Science.

A third paper has been generated from USML-1 sequences of oscillation and natural decay. The experimental results were obtained using rotating and non-rotating drops with small but finite viscosities. The latter data agree with current theory while the flattening inherent in a rotating drop caused deviation from the simple models. This work has been submitted to the Journal of Fluid Mechanics.

A major reason that the science return from USML-1 was less than expected was the appearance of an uncontrollable rotation. In support of the DPM Project a team at Vanderbilt has been studying how the air moves inside the DPM chamber under various combinations of acoustic signals. Flows were observed to be due to both to the quartz wind from the individuals drivers as well as to the interaction of DPM's unique signals. The strength of the former is larger and correlates with the weak DPM driver on USML-1. Characterization measurements of the torque on a suspended ball in DPM-like chambers at Vanderbilt, the trainer at Marshall SFC, and the flight system at KSC are being carried out.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	3	MS Degrees:	2
PhD Students:	1	PhD Degrees:	0

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TASK INITIATION: 8/92 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 963-24-04-01

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Lee, C.P. and Wang, T.G. Acoustic radiation pressure. J. Acoust. Soc. Am., vol. 94, 1099-1109 (1993).

Wang, T.G., Anilkumar, A.V., Lee, C.P. and Lin, K.C. Core-centering of compound drops in capillary oscillations: Observations on USML-1 experiments in space. Journal of Colloids and Interface Science, vol. 165, 19-30 (1994).

#### Proceedings

Wang, T.G., Anilkumar, A.V., Lee, C.p. and Lin, K.C. "AIAA Paper No. 93-0252, (1993)." A preliminary analysis of the USML-1 drop dynamics experiment results.

*Colloid Physics in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. David A. Weitz

EXXON Research and Engineering Co.

Co-INVESTIGATORS:

Prof. P.N. Pusey

The University of Edinburgh

**TASK OBJECTIVE:**

This experiment entails the study of the physics of colloidal particles in microgravity. It consists of two distinct parts. The first deals with ordered structures while the second deals with highly disordered structures. The study of ordered structures entails the growth of colloidal superlattices formed with mixtures of different-sized particles. The goal is to develop useful periodic structures using colloidal particles as precursors, through "colloid engineering." The study of the highly disordered structures entails the formation of fractal colloidal aggregates of much greater extent than has ever been done, and the formation of very weak structures that would collapse under their own weight in normal gravity.

**TASK DESCRIPTION:**

The work within this effort will be ground-based research to study the formation of novel materials from colloidal dispersions, and to study the physical properties of such materials. As part of the effort, space experiments to be carried out in a space shuttle middeck carrier, will be defined. These experiments will utilize the laser light scattering apparatus currently being developed at NASA LeRC.

The focuses of the ground-based experiments will be the study of colloidal superlattices formed from mixtures of different-sized colloidal particles, the in-depth study of the formation of fractal colloidal aggregates, and the study of granular particles fluidized in a gas. While considerable knowledge exists about the formation or growth of fractal colloidal aggregates, much less is known about the unique properties of these objects and the consequences of their scale invariance. A major reason for this is the relatively small scale over which the aggregates exhibit scale invariant behavior. By growing structures that are scale invariant over a much greater range of length scales, the properties of these objects can be studied much more directly. This will provide the first detailed information about the consequences of scale invariant structure on the properties of these materials.

**TASK SIGNIFICANCE:**

Very little is currently known about the structures of binary colloidal crystals, and these experiments will initially be directed at determining the phase diagrams of the superlattices for mixtures of different sizes of particles. In addition, virtually nothing is currently known about the kinetics of the formation of these superlattices, and about their dynamics once they are formed. This will also be studied by these experiments. This will represent the first in-depth study of the growth and properties of colloidal superlattices.

**PROGRESS DURING FY 1994:**

The necessary arrangements for the grant were put in place in June 1994. The SCR will take place on October 28, 1994. The investigators have developed plans for the experiments to be conducted in space. This plan will be presented at the SCR. The ground-based research to support the flight experiment has just been initiated.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 3/94 EXPIRATION: 3/97

PROJECT IDENTIFICATION: 963-24-05-13

RESPONSIBLE CENTER: LeRC

*Study of Two Phase Flow Dynamics and Heat Transfer at Reduced Gravity*

PRINCIPAL INVESTIGATOR: Prof. Larry Witte

University of Houston

CO-INVESTIGATORS:

J.B. McQuillen

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objective of this study is to develop and experimentally verify theoretical models that predict gas-liquid flow regimes and their characteristics in reduced gravity.

**TASK DESCRIPTION:**

Reduced-gravity experiments will be conducted in NASA aircraft to measure the previously listed two-phase flow parameters for a range of tube diameters, gas and liquid flow rates, and fluid properties. The gas phase for the experiments will be air; the liquids to be employed are water, water-glycerin mixtures, and water-zonyl mixtures. A theoretical modeling effort will be integrated with the experimental efforts.

**TASK SIGNIFICANCE:**

The purpose of this study is to achieve a better understanding—better predictability—of two-phase (gas-liquid) flow in pipes to assist in the design of space-based power and thermal management systems and of the terrestrial-based nuclear power plants and oil and natural gas pipelines.

**PROGRESS DURING FY 1994:**

A series of low gravity tests were conducted aboard the NASA LeRC Learjet in November and December 1994 using a 1.27 cm. inner diameter test section. The focus of these tests was to obtain additional data concerning the characteristics of slug and annular flow. A new technique using a hot film anemometer to measure wall shear stress was incorporated.

In February, the original PI for this effort, Dr. Abe Dukler passed away. The University of Houston, with the concurrence of the NASA technical grant monitor, named Dr. Larry Witte to succeed Dr. Dukler. NASA Headquarters decided to descope the original plans for a space flight experiment into a ground-based activity.

In April 1994, several tests were performed aboard the NASA JSC KC-135 yielding flow regime maps for air and three fluids -- water, water-glycerin, and Zonyl-FSP/water in a 2.54 cm. inner diameter tube. Additional measurements were made, both near the gas-liquid mixer and near the exit, of the liquid film thickness, void fraction, pressure drop and wall shear stress.

A 2.54 cm. inner diameter heated test section was designed and fabricated to extend the flow dynamics research into microgravity two-phase heat transfer. The section is designed to obtain local, time-averaged heat transfer coefficients along the heated length as well as instantaneous coefficients that can be correlated with the motion of liquid waves or slugs. Resistance temperature elements (RTD's) are positioned between the heaters and the external tube to estimate the internal wall temperature. A thin-film thermocouple, capable of response times as short as 103 milliseconds, is mounted inside the tube near the exit. A film thickness probe just after the exit is used to correlate the temperature fluctuations with waves or slugs. A special mixer section is used after the heated test section to obtain the mixing-cup temperature.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-24-0A-35

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

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Bousman, W.S. and Dukler, A. "Ground based studies of gas-liquid flows in microgravity using learjet trajectories." AIAA paper 94-0829, presented at AIAA Aerospace Sciences Meeting, Reno, January 1994.

Bousman, W.S. and McQuillen, J.B. "Characterization of annular two-phase gas-liquid flows in microgravity." presented at the Second Microgravity Fluid Physics Conference, Cleveland, OH, January 1994.

*In Situ Monitoring of Crystal Growth Using MEPHISTO*

PRINCIPAL INVESTIGATOR: Prof. Reza Abbaschian, Ph.D.

University of Florida

CO-INVESTIGATORS:

A.B. Gokhale

University of Florida

S.R. Coriell

National Institute of Standards and Technology (NIST)

J.J. Favier

CENG (France)

**TASK OBJECTIVE:**

The objective is to determine the morphological stability of solid/liquid interfaces and resulting macro and micro segregation patterns, and to determine the attachment kinetics at the freezing interface deduced via measurements of the growth-rate/interface-supercooling relationship.

**TASK DESCRIPTION:**

To investigate the solidification behavior and stability of solid/liquid interfaces during the growth of pure Bi (a facet forming material), and Bi alloyed with small amounts of Sn, in 1-g and  $\mu$ g. The experiments were designed to make use of the second flight of MEPHISTO on USMP-2 (3/94).

The experiments make use of the Seebeck technique to measure the interface temperature in-situ and non-invasively during crystal growth in both the ground-based and flight experiments. Both 1-g and  $\mu$ g experiments make use of the measured resistance change across the sample to determine interfacial velocity and Peltier pulsing for demarcation of the interface shape.

**TASK SIGNIFICANCE:**

The first flight studied morphological stability in Sn alloyed with Bi (non-facet forming material), conducted by French scientists as part of this collaborative study. The results of the second flight experiment will therefore complement the results and findings of the first flight.

**PROGRESS DURING FY 1994:**

A comprehensive directional solidification experiment was recently carried out successfully on the USMP-2 mission (STS-62) utilizing the MEPHISTO directional solidification facility. The 14 day shuttle flight was launched on Friday, March 4, 1994. Using three samples processed in parallel, a total of 45 cm of dilute Bi-Sn alloys were solidified directionally in microgravity under well controlled and well characterized conditions. Prior to the final directional solidification, extensive measurements were performed on the samples, consisting of Seebeck measurements to measure the solid/liquid (s/l) interface temperature, resistance measurements to track the position of the s/l interface and thermal gradient measurements in the solid and liquid during freezing and melting. The final solidification also included a procedure for marking the shape of the s/l interface via mechanical perturbations, as well as rapid quenching of a 2 cm section of one of the samples.

The experiments were performed to gain a detailed understanding of the role of gravity driven convection during the solidification of faceted materials. Two fundamental and interrelated aspects of the liquid to solid transformation have been investigated: (a) Morphological stability of the solid/liquid (s/l) interfaces and the resulting macro- and micro-segregation patterns and (b) atomic attachment kinetics at the freezing interface, deduced via measurement of the growth rate-interface supercooling relationship(s)

To achieve the goals of the project in a relatively short period, a three-pronged strategy for achieving the stated goals was used: (a) Development of experimental apparatus for ground based kinetics and morphological stability studies at University of Florida (UF), (b) Scientific and technical collaboration with the MEPHISTO teams at CENG and

CNES and (c) Analytical modeling of morphological stability and interface kinetics in collaboration with Sam Coriell (NIST). Particular reference is also made to an extensive collaboration between the various scientific and technical personnel from NASA-Lewis for developing a comprehensive flight program.

The research team at UF developed the facilities necessary for ground based experiments to ensure maximum conformity with the MEPHISTO space hardware. In addition, four "campaigns" were conducted prior to the USMP-2 mission (three on the MEPHISTO engineering model at CENG and one on the MEPHISTO flight model at CNES). Each campaign required three samples approximately 1 meter in length, which were prepared at UF according to MEPHISTO specifications. The campaigns not only proved the integrity of the samples produced, but also provided valuable ground based data, which is currently being compared with the flight experiments. Concurrently, NIST has carried out analytical modeling of the morphological stability of faceted solid/liquid (s/l) interfaces for the alloy system under investigation.

These flight experiments utilized a novel technique (termed the Seebeck technique) to measure the interface supercooling directly, non-invasively and in-situ (i.e. in real time during growth). The interface velocity was measured by monitoring the resistance change across the sample, while the interface shape was delineated by subjecting the sample to electrical current pulses (for ground based studies) and mechanical perturbations (for  $\mu$ -g studies) to cause a momentary demarcation of the interfaces.

Initial ground-based experiments were carried out using high purity Bi and dilute Bi-Sn alloys. Bi-Sn alloys were chosen to complement the experiments conducted by CENG/CNES on the first flight of MEPHISTO (MEPHISTO-1): on this flight, dilute, non-faceted Sn-Bi alloys were used, while this research program (the second flight of MEPHISTO, or MEPHISTO-2) used strongly faceted Bi- 0.1 at % Sn alloys. In this manner, the results of the two flights are being used to compare and contrast various fundamental aspects of solidification without and with a strong influence of atomic attachment kinetics, respectively, in the presence (ground-based studies) and near-absence ( $\mu$ -g studies) of gravity induced convection. It is expected that this comprehensive investigation approach will significantly further our understanding of key crystal growth parameters.

We further expect to use these data to test and improve many of the current solidification theories. In particular, the interplay between morphological stability and interface kinetics is not well understood at the present time. The microgravity experiments will yield an integrated database involving interface velocity/interface shape/interface supercooling. Such data are important from both practical and theoretical standpoints. For example, a knowledge of the transition from a faceted to a rough interface (from the Seebeck data) and the interface shape (from solute-dump-demarcated interfaces) under identical growth conditions has important applications in practical crystal growth situations: the information can be used to understand the correlation between defect generation and solute banding. In addition, because the information has been obtained in diffusion dominated conditions without the overriding effects of gravity-induced thermo-solutal convection, meaningful tests (and appropriate refinements) of the current crystal growth theories can be made.

Two other spin-offs of the proposed program are worthy of note here: we will be able to obtain values of key parameters, such as liquid diffusivities, via this investigation. In addition, the novel and non-intrusive technique used to measure the interface temperature can potentially be utilized for monitoring and controlling the space-based single crystal growth of technologically important semiconductors.

We are currently analyzing the approximately 6 gigabytes of USMP-2 MEPHISTO data. During the mission, extensive use was made of the telemetry commanding capability to modify and refine experimental procedures for better scientific yield. Preliminary analysis of the data acquired during the first 28 hours of mission shows excellent correlation of the Seebeck signal with melting/freezing as well as solute build-up/decay. Numerical calculations are being carried out concurrently to correlate the Seebeck signal with thermal/solutal decay and hence to back calculate an accurate value of the diffusion coefficient of tin in liquid bismuth.

## II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 4  
PhD Students: 0

TASK INITIATION: 1/90 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 963-25-05-04

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Abbaschian, R., Gokhale, A.B., DeWitt, R., Cambon, G., Favier, J.J., Coriell, S.R., and deGroth, III, H.C. "In situ monitoring of crystal growth using MEPHISTO." Proceeding of the Microgravity Materials Science Conference, Huntsville, Alabama, May, 1994.

Gadonniex, D., Gokhale, A., and Abbaschian, R. "Morphological stability of faceted solid/liquid interfaces in dilute Bi-Sn alloys." Proceedings of the 32nd Aerospace Science Meeting, AIAA 94-0791, January, 1994.



*Coupled Growth in Hypermonotectics*

PRINCIPAL INVESTIGATOR: Dr. J. B. Andrews

University of Alabama, Birmingham

CO-INVESTIGATORS:

A. Sandlin

National Research Council (NRC)

S. Coriell

National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE:**

The objective of this investigation is to gain a further understanding of solidification processes in immiscible alloy systems. A portion of the study involves the development of a model for the coupled growth process in monotectic systems. This analysis starts with the basic equations for diffusion controlled growth and avoids many of the simplifying assumptions often utilized in similar analyses. A parallel effort is underway to develop and refine experimental techniques which will permit steady-state coupled growth of hypermonotectic composition samples to produce aligned microstructures. The results from these experiments will then be compared to predictions from the model and utilized to improve the model. In order to permit steady-state coupled growth in hypermonotectic composition samples experimentation must be carried out under low-gravity conditions.

**TASK DESCRIPTION:**

This project includes the major research tasks of theoretical modeling, selection of sample materials, selection of ampoule materials, experimentation, data analysis, and development of ampoules for processing. In addition, the project is concerned with the evaluation of current flight hardware for use in experimentation and with input into the development of new hardware. Experimentation requires directional solidification of immiscible aluminum-indium alloys under low-gravity conditions in order to avoid convective instability and promote steady-state coupled growth.

**TASK SIGNIFICANCE:**

The significance of this project lies primarily in the scientific gains to be made in truly understanding the coupled growth process in immiscible alloys. Many alloys in immiscible systems have great promise for potential applications in areas which include superconductors, magnetic materials, catalysts, and electrical contacts. However, there are many details of the solidification process that are poorly understood for these alloys because these details are masked by gravity driven phenomena. This project is aimed at using the unique environment available in space to improve this understanding in order to make possible the production of new materials using specialized processing techniques.

**PROGRESS DURING FY 1994:**

Major advances have been made in the development of a model for diffusion- controlled coupled growth in monotectic systems. Starting with the basic equations, the model has been developed without many of the simplifying assumptions usually made in this area. We have found that some of these presumably valid assumptions lead to a major restriction on the phase equilibria conditions for which the previous analyses are valid. These restrictions are avoided with the newly developed model. In addition, the new model determines the volume fractions of the phases in a self-consistent way. This factor may be quite significant at higher growth velocities.

In the experimental area, contact angle and chemical compatibility tests have led to the selection of AlN and sintered  $\text{Si}_3\text{N}_4$  as viable ampoule materials for microgravity experimentation. Flight ampoule designs are nearing the testing stage. Work has been carried out which validates the current model for morphological stability conditions in hypermonotectic Al-In alloys. In addition, compositional analysis of ground processed hypermonotectic Al-In samples indicates convective instability is a major factor impeding the establishment of steady-state growth conditions. This ground based work has permitted the establishment of processing limits for microgravity experimentation on the Al-In alloy system.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 963-25-08-09

NASA CONTRACT NO. NAG8-39717

RESPONSIBLE CENTER: MSFC

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*Effects on Nucleation by Containerless Processing*

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PRINCIPAL INVESTIGATOR: Prof. Robert J. Bayuzick

Vanderbilt University

## CO-INVESTIGATORS:

W. Hofmeister

Vanderbilt University

M. Robinson

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

The primary scientific objective is to further the understanding of nucleation of solids from their melts. A secondary objective is to determine if ground based methods, such as drop tube processing, electromagnetic levitation, and electrostatic levitation, are equally useful for containerlessly processing bulk samples of pure metals as compared to electromagnetic heating and positioning in low Earth orbit. Within this secondary objective is a focus on identifying and quantifying any possible technique specific factors that influence nucleation behavior.

**TASK DESCRIPTION:**

The presently existing containerless ground based methods are being used to study nucleation of solid from the liquid. This includes drop tube processing, electromagnetic levitation, and electrostatic levitation. Since nucleation is a statistical process, approximately 100 undercooling measurements are desired for each type of sample. This number of measurements facilitates the interpretation of results through the application of statistical techniques. Much care is taken in the measurement of temperature due to the sensitivity of the approach to the precision of the measurements. Increasing and investigating the absolute precision is an important part of the experiments. The precision affects the width of the distribution of undercoolings, which consequently determines the activation energy for the phenomena. A large half-width of a distribution yields low values for the preexponential and exponential factors in the nucleation equation, thereby indicating heterogeneous nucleation by mechanisms other than contact with a container. Comparisons of the data from the ground based and flight techniques give clues as to the nature of the nucleation of the solid from the liquid. Different processing methods have different environments and other factors that may affect the amount of undercooling in bulk samples.

**TASK SIGNIFICANCE:**

Solidification processing is one of the most prominent methods for the production of materials and most of these processes begin with a nucleation step. The regime of nucleation known as deep undercooling, where liquids are cooled considerably below their equilibrium freezing temperatures prior to the formation of solids, has become particularly distinctive. With deep undercooling, a unique condition for microstructural development and control exists and, therefore, a unique condition for improving and controlling the properties of materials exists. Hence, an understanding of the rudiments of nucleation in the unique regime is most important.

**PROGRESS DURING FY 1994:**

Results indicate fundamental differences in the nucleation behavior of the same sample types in the three ground based processing techniques, drop tube processing, electromagnetic levitation, electrostatic levitation. For zirconium, which has been processed by all three techniques, the distributions of undercoolings are all non-Gaussian and clustered around the same undercooling but are wider for the electromagnetic levitator and the drop tube than for the electrostatic levitator. With all three techniques, the distributions retain the same general appearance but shift their centers to higher undercoolings (lower nucleation temperatures) as the material purity is increased from 99.8% to 99.95%. This trend toward higher overall undercoolings but similar distributions was also observed for 99.995% pure zirconium samples processed in the electromagnetic levitator. However, the 99.995% purity material did not undercool as well in the drop tube as it did in the electromagnetic levitator, although the shape of the distribution was similar. For niobium, processing to date has been accomplished only in the drop tube. In this case

distributions were much narrower than for zirconium although the percentage undercooling for similar purity levels was approximately the same.

Due to the presence of ultra high purity helium in the electromagnetic levitator, the cooling rate of the samples can be changed by adjusting the flow of cooling gas. Two sets of undercoolings, one obtained at a cooling rate of 100 K/sec and the other at 20 K/sec, have been produced for the same sample type. The shape of the distributions was the same, but the higher cooling rate set of undercoolings was clustered around an undercooling 10 K higher than the center of the lower cooling rate set.

The values of the preexponential factors and the activation energies in the classical nucleation rate equation affect the distributions of undercooling data. A set of undercoolings distributed over a larger range has lower values, whereas a set of undercoolings distributed over a narrower range has higher values associated with it. Thus, for zirconium, the drop tube and electromagnetic levitator had lower associated values than did the electrostatic levitator. The preexponential factors and activation energies tend to be higher as the undercoolings shift to higher overall values. With zirconium, values for the preexponential factors and activation energies ranged from 108 to 1013 and 13 kT to 24 kT, respectively. For niobium, these values were 1034 and 72 kT. Overall, the values obtained for niobium much more closely approach the values predicted for homogeneous nucleation as calculated by Turnbull and Fisher using classical nucleation theory.

The flight experiment in low Earth orbit using TEMPUS, an electromagnetic heater and positioner, resulted in only one undercooling measurement for zirconium. This undercooling was 160 K. Due to sample instability in the facility, the experiments were prematurely terminated when the samples welded to the cage. However, even though no results on the nucleation behavior of zirconium were possible due to the lack of data, thermophysical property measurements can be obtained. The latent heat of fusion of zirconium was determined to be 14.5 kJ/mol whereas values reported in the literature to date vary between 14.5 and 16 kJ/mol. In addition, the large surface oscillations that occurred while the sample was molten should enable the determination of the surface tension and viscosity of zirconium in the undercooled state. Surface analysis of the samples is being conducted using Auger Electron Spectroscopy (AES) and Electron Spectroscopy for Chemical Analysis (ESCA) in order to determine the chemical compositions of the surfaces of the samples.

Preparations and additions were made in order to ready MEL, another electromagnetic heating and positioning facility, for flights in the NASA KC-135 aircraft. Nucleation frequency experiments using MEL, will be conducted in vacuum at a level of around  $10^{-6}$  torr. The results will complement and allow comparisons between the ground based techniques.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 4/90 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-25-08-06

NASA CONTRACT NO.: NAG8-978

RESPONSIBLE CENTER: MSFC

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*Alloy Undercooling Experiments in Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. Merton C. Flemings

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

D.M. Matson

Massachusetts Institute of Technology (MIT)

**TASK OBJECTIVE:**

The objectives of this task are to perform solidification experiments on undercooling binary alloys, to compare results of ground-based and microgravity experiments, and to examine effects of microgravity on solidification behavior and microstructure characteristics.

**TASK DESCRIPTION:**

Through experiments applying direct, high speed, high resolution pyrometric and cinematographic measurements during melting, undercooling and recalescence of nickel-tin binary alloys of different compositions, both on the ground and in microgravity, collect thermal history, nucleation and growth history, and resulting solidification microstructures.

**TASK SIGNIFICANCE:**

With experiments carried out in microgravity, it is expected to have improved specimen shape and stability and reduced convection during cooling, resulting in the possibility of higher undercooling, less microstructure alteration, reduced coarsening, and improved specimen observation in order to gain a complete understanding of the solidification kinetics of undercooled melts, including: primary dendrite tip velocities; rapid thickening of primary and secondary arms during recalescence; ripening, remelting, and solute redistribution; dendrite fragmentation and grain refinement; primary phase solidification and ripening; and eutectic solidification with concurrent primary phase ripening.

**PROGRESS DURING FY 1994:**

Work during the current grant period has resulted in the successful completion of the preliminary preparations for the flight undercooling experiments and support of the IML-2 TEMPUS flight investigations. Samples have been prepared, analyzed, and submitted for installation in the flight module. Preliminary investigations to define optimal processing parameters were completed utilizing the NASA KC-135 experimental platform. All flight samples were subsequently processed by melting and observing the solidification behavior in microgravity during STS-65 and are presently under analysis following recovery from the orbiter payload compartment. Sample metallography and experiment documentation are also currently in progress.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 4/90 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-25-05-03

NASA CONTRACT No.: NAG8-971

RESPONSIBLE CENTER: MSFC

*Compound Semiconductor Growth in Low-g Environment*

PRINCIPAL INVESTIGATOR: Dr. Archibald L. Fripp

NASA Langley Research Center (LaRC)

CO-INVESTIGATORS:

R.K. Crouch

NASA Headquarters, Code UG (MSAD)

W.J. Debnam

NASA Langley Research Center (LaRC)

I.O. Clark

NASA Langley Research Center (LaRC)

**TASK OBJECTIVE:**

The objective of the Langley flight program is to determine the effects of gravity driven convection on the growth and crystal properties of the compound semiconductor alloy, lead tin telluride which is miscible over the entire compositional range. The electronic properties of this material are dependent on the ratio of the two, pseudobinary, components and consequently, the uniformity of an array of devices is dependent on good compositional control. Lead tin telluride is amenable to study for it is easily compounded; it has a relatively low vapor pressure; and there is existing, though limited, literature on its growth and properties.

**TASK DESCRIPTION:**

This material was chosen for microgravity research for a number of reasons. Lead tin telluride is not only a useful semiconductor material which has been used for construction of infrared detectors and tunable diode lasers. It also has a phase diagram similar to other compound semiconductors of interest such as mercury cadmium telluride and mercury zinc telluride.

Lead tin telluride is also interesting from a purely scientific point of view in that it is both solutally and thermally unstable. Both the temperature gradients and the compositional changes in the liquid near the melt/solid interface produce density gradients which, in turn, produce driving forces for convection when coupled with gravity.

**TASK SIGNIFICANCE:**

Earth based Bridgman growth of lead tin telluride has only produced inhomogeneous crystals that are a result of strong convective forces in the liquid during growth. The temperature gradients are required for growth and the solutal changes at the interface are a fundamental property of the material system. However, for convection to occur these gradients must be coupled to a gravitational field. Growth in low Earth orbit offers an unique and fascinating opportunity to study the effect of convection on this class of materials. The resultant gravitational force is not zero in low Earth orbit hence convection is not completely eliminated but the fluid velocity, due to convection, will be greatly reduced.

Two flights are planned in the Advanced Automated Directional Solidification Furnace (AADSf). The primary objective of both flights is to study the effect of gravity reduction, hence convection reduction, on the growth of lead tin telluride. In one experiment the growth rate of the crystal will be changed in steps to test the effect of varying the relative speed of the interface movement and the fluid velocity. In the other experiment the Space Shuttle will be rotated to vary the relative orientation of the gravity vector and the crystal growth axis. Both sets of experiments are expected to affect the compositional homogeneity of the crystal.

**PROGRESS DURING FY 1994:**

This years effort has continued on finalizing the AADSf furnace configuration and calibration. Lead tin telluride has been grown in the flight prototype furnace. The newly designed calibration cartridge has been used for extensive characterization of the AADSf furnace. Doped germanium samples, with interface demarcation, have been grown in the prototype AADSf for comparison to the calibration data. Numerical modeling of the furnace and samples has

shown remarkable correlation with experimental measurements on calibration samples and the marked germanium samples.

A cartridge with a miniature, internal, pressure gauge was designed and tested in furnaces both at Langley and Marshall. These tests not only measured the pressure as a function of set point temperatures over the different thermal gradients but also measured the integrity of the cartridge seals and the rigidity of the cartridge as a function of temperature and differential pressure. Both the inconel and WC-103 cartridges have been evaluated.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	0
PhD Students:	3	PhD Degrees:	1

TASK INITIATION: 10/78 EXPIRATION: 9/98

PROJECT IDENTIFICATION: 693-80-07-01

RESPONSIBLE CENTER: LaRC

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*Melt Stabilization of PbSnTe in a Magnetic Field*

PRINCIPAL INVESTIGATOR: Dr. Archibald L. Fripp

NASA Langley Research Center (LaRC)

CO-INVESTIGATORS:

W.J. Debnam

NASA Langley Research Center (LaRC)

F.R. Szofran

NASA Marshall Space Flight Center (MSFC)

A. Chait

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objective of this research is to further elucidate the gravity driven physical phenomena on the growth of the alloy compound semiconductor, PbSnTe. This work, coupled with the past microgravity experiment with the MEA and the existing flight program to grow PbSnTe in the AADSF, will form the most comprehensive set of space processing experiments performed to date.

**TASK DESCRIPTION:**

The effect of the gravitational body force on the convective properties of the alloy compound semiconductor, PbSnTe, with that body force modified by both reduced gravity and by magnetohydrodynamic (mhd) damping is the subject under investigation. PbSnTe is an ideal material for this study in that it was the material of both a past flight experiment and a planned 1996 AADSF experiment. Both of these experiments are without magnetic fields. Subsequent experiments, both Earth based and in Space, using mhd damping will form a complete set of experiments that will further elucidate the gravity dependent physical phenomena on the growth of this class of materials.

The application of a magnetic field to PbSnTe growth will dampen convective flow. The anticipated results are that even in the MSFC superconductor magnetic furnace the growth will not become diffusion controlled but that the combination of magnetic field and low gravity environment will produce diffusion controlled growth.

**TASK SIGNIFICANCE:**

Numerical modeling is an integral part of this endeavor. Computer simulation can aid in the design of the space experiment by its predictive capacity to optimize conditions for the growth. The key purposes of this portion of the study will be to optimize the growth for both the Earth and the space experiments and to obtain an estimate of the required magnetic field strength for low gravity growth.

This proposed work will complete the set. It will compare the effects of convection, as modified by a magnetic field, on the growth of this material both on Earth and in the Microgravity environment found in low Earth orbit.

**PROGRESS DURING FY 1994:**

The progress within this first year of the research on the magnetic stabilization of PbSnTe consists primarily of preparations for future quantifiable, modelable experiments.

Numerical modeling is an essential part of this program. The primary thrust for modeling is within the Materials Division at the Lewis Research Center. Modeling, both two and three dimensional, will evaluate both the sensitivity of the thermophysical parameters to determine if better measurements, in addition to electrical resistivity, are needed and attempt to determine the combination of low gravity and magnetic field strength required to attain diffusion controlled growth in PbSnTe. Preliminary modeling is complete. Diffusion controlled growth is predicted, with modest magnetic fields, in microgravity but heavy mixing is predicted on Earth even with a five Tesla field.



The ampoule configuration has been designed for ground based tests in the five Tesla magnetically stabilized furnace at the Marshall Space Flight Center. Seven crystals have been grown to date, six with full magnetic field and one with no field. The primary variable was ampoule pull rate. All crystals showed complete compositional mixing as was predicted by the numerical modeling.

Additional modeling is underway to evaluate the ampoule diameter as it affects both viscous damping and extent of flux line intersection. The effect of thermal gradient, both magnitude and shape, will also be evaluated.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 10/93 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 963-80-07-05

RESPONSIBLE CENTER: LaRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Fripp, A.L., Debnam, W.J., Szofran, F.R., and Chait, A. "Melt stabilization of PbSnTe in a magnetic field." Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

*Gravitational Role in Liquid-Phase Sintering*

PRINCIPAL INVESTIGATOR: Prof. Randall M. German

Pennsylvania State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The purpose of this research is to establish the gravitational role in liquid phase sintering with respect to both the macro-scale distortion and phase separation, and the microstructural evolution over time at elevated temperatures.

**TASK DESCRIPTION:**

The investigation has graduated to flight status, with 21 samples (seven compositions of tungsten-nickel-iron) subjected to liquid phase sintering treatments on STS-65 during July 1994. The experiments involved isothermal hold times of 1, 15, and 120 minutes at 1500°C in the Japanese-developed Large Isothermal Furnace on the IML-2 mission. Identical ground-based experiments have been conducted to provide the baseline for contrast with the microgravity samples, and post-flight analysis will focus on quantitative assessment of distortion and microstructural evolution.

**TASK SIGNIFICANCE:**

The microgravity experiments will establish a modeling basis for the gravitational role in a viscous flow distortion of sintered components. It will further assess the agglomeration of solid grains and possible coalescence contributions to grain growth for upgrading of current theories. These results will lead to the development of manufacturing techniques which will permit formulation of new unique alloys.

**PROGRESS DURING FY 1994:**

"Sintering" means welding or fusing of metal or ceramic powders by heating them without melting. Frequently, it is aided by applying pressure in a special high-temperature press to squeeze the particles together. This experiment explores a different mechanism, by adding a portion of a powder that melts at a lower temperature and surrounds the powders that remain solid. This liquid then lets particles and materials move more easily, allowing the powders to more rapidly form a solid compact. Problems such as separation of the solid and liquid due to gravity (manifested by settling of the solid particles) still remain. Access to the low gravity environment of Earth orbit provides a unique opportunity to study liquid-phase sintering without separation, settling, or other gravity-induced complications. The results will increase our understanding of the process, and the new knowledge will help introduce new industrial application of liquid-phase sintering, such as automotive components, resulting in new and improved products.

In this investigation, mixed powders of tungsten, nickel, and iron are initially cold compacted under pressure in the shape desired for the final product. The compacts are then heated to just below the nickel-iron alloy melting temperature to provide handling strength, a process called "presintering." In the experiment, they are heated above 1465°C to form a liquid-solid mixture. The tungsten, with its very high melting point (3370°C), remains a solid, while the nickel and iron, with much lower melting points, become liquid. The liquid permits more rapid transport of material for faster sintering than would be possible if all the material were solid. After sintering, the microstructure of the samples (i.e., the structure when viewed under very high magnification), consists of connected tungsten grains surrounded by the solidified liquid.

This experiment flew as part of the second International Microgravity Laboratory (IML-2) mission in July 1994 aboard the Space Shuttle Columbia. The experiment was conducted in an apparatus called the Large Isothermal Furnace (LIF), which could operate at the high temperatures required. The LIF was developed by Ishikawajima-Harima Heavy Industries Co., Ltd., for the National Space Development Agency of Japan (NASDA).

This project was supported by the NASA Headquarters Microgravity Science and Applications Division, and was managed by the NASA Lewis Research Center.

The test specimens for the LIF consisted of three different cartridges, each containing seven samples 10 mm in diameter by 10 mm high. One cartridge was tested at each of the three critical sintering periods identified in earlier ground-based experiments: 1, 15, and 120 minutes. In one minute, liquid penetrates along existing solid-solid boundaries. Fifteen minutes is the time needed for full densification. The 120-minute time is needed to observe grain rotation and coalescence events.

In summary, a nominal temperature of 1506°C was achieved for each of the desired test times. Free-drift of Columbia during the appropriate times was confirmed. All of the functional objectives were achieved. Analysis will begin upon return of the samples to the laboratories of the Pennsylvania State University.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 10/94 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-25-05-05

RESPONSIBLE CENTER: LeRC

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*Isothermal Dendritic Growth Experiment*

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PRINCIPAL INVESTIGATOR: Prof. Martin E. Glicksman

Rensselaer Polytechnic Institute

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

Successfully conduct the Isothermal Dendritic Growth Experiment (IDGE) on the STS-62 Space Shuttle mission. Analyze resulting space flight and terrestrial data to determine dendritic tip velocity and tip radius as functions of supercooling (temperature). Use velocity and radius data to evaluate the validity of theoretical models, some of which are nearly 50 years old, that purport to predict velocity and radius (these models are used to improve industrial metal production on Earth). Publish reports and give papers to inform the scientific community of our findings.

**TASK DESCRIPTION:**

The Isothermal Dendritic Growth Experiment (IDGE) is a fundamental Materials Science experiment performed on the Space Shuttle. The specific topic is dendritic solidification which is relevant to virtually all industrial manufacture of metals and alloys on Earth.

IDGE will be performed in the cargo bay of the Space Shuttle using an apparatus designed, built, and tested at the NASA Lewis Research Center (LeRC) in Cleveland, Ohio, USA. Over 400 photographs of dendrites that solidified in space along with over 800 photographs of dendrites solidified on Earth will be produced on the first flight (STS-62). Each photograph will be accompanied by 8 (or more) scientifically important measurements of time, temperature, and local acceleration. While in space, IDGE will be operated by LeRC personnel located in the Payload Operations Control Center (POCC) at the George C. Marshall Space Flight Center (MSFC) in Huntsville, Alabama, USA.

In the months prior to the flight, we will make several trips to LeRC and MSFC for mission operations planning and training. In addition, at RPI, we will analyze terrestrial data produced by the IDGE flight apparatus prior to its turn over to Kennedy Space Center (KSC) personnel on July 28, 1993.

We will work with the LeRC personnel during the flight to assess the IDGE Slow Scan Television (SSTV) dendrite images in real time and replan subsequent experiments to take maximum advantage of circumstances.

After the flight, we will analyze the resulting 35mm photographs and other data to determine the dendrite tip velocity and dendrite tip radius as functions of supercooling. These will be compared to predictions made by many theoreticians over the last 50 years (these theories have been used to try to improve industrial metal production on Earth).

As soon as possible thereafter, we will publish reports and give papers at relevant conferences to disseminate the information to the scientific and industrial community.

**TASK SIGNIFICANCE:**

Ultimately, IDGE may result in improved industrial manufacturing of steel, aluminum, super alloys, and other metals that we use on Earth every day. Moreover, the data returned from space will remain relevant indefinitely. In fact, the Schriffer Committee report (1987) declared that IDGE was one of NASA's four "world class" microgravity science experiments due to the quality, scope, and long term relevance of its science.

IDGE will study Dendrites. Dendrites are tiny crystalline structures that form inside molten metal alloys when they solidify during manufacture.

In summary IDGE is significant because:

Dendritic growth is the ubiquitous form of crystal formation encountered when metal alloys and many other materials solidify. Most industrially important metals (\$Billions) solidify dendritically from the molten state.

Examples: Cast iron alloys and cast aluminum alloys used in automobile engine blocks.

The dendrites formed inside alloys during manufacture result in microscopic zones of strength, weakness, ductility, and brittleness. Consequently, understanding precisely how dendrites form can lead to improvement of alloy strength and ductility by eliminating weak and brittle areas in the metal. Production costs can also be lowered.

IDGE provides the only unambiguous set of scientific data that can be used to test and correct the theoretical models of dendritic formation used to improve alloy production. Data gathered on Earth are confounded by gravitationally induced convective effects.

During the past 50 years, numerous scientists have attempted to develop practical theoretical models to predict important dendritic growth parameters. However, years of experimentation on Earth has not produced a dataset capable of testing the models to find out which one is correct (if any). This is due to the influence of gravity.

On Earth, dendritic solidification can be strongly affected by gravitationally driven convective currents in the molten metal. These currents exist during virtually all industrially important metal solidification processes. However, their effect on dendritic solidification cannot be accurately modeled without knowledge of dendritic solidification in the absence of such currents.

The microgravity environment in space effectively turns off convection in the IDGE experiment. Extensive non-advocate peer review indicated that a comparison between space experiment data and Earth experiment data is the only practical way to separate the effects of convection from the underlying mechanisms of dendritic solidification.

#### **PROGRESS DURING FY 1994:**

The Isothermal Dendritic Growth Experiment (IDGE) is a tele-operated microgravity science experiment payload that flew on the space shuttle Columbia STS-62 mission during March 1994. It was an outstanding success.

All preflight activities were conducted as planned. These included evaluation of terrestrially acquired IDGE data and six trips to LeRC and MSFC for planning and training for flight operations.

During flight, teleoperation allowed us (RPI and LeRC personnel) to modify the experiment protocol and, thereby, take advantage of unexpectedly fortuitous circumstances. Consequently, by one measure, IDGE obtained nearly 300 percent of the required data. Specifically: 58 dendrites were grown versus 20 required (290% success); the supercooling (independent variable) dynamic range spanned 37 to 1 versus 10 to 1 required (370% success); resulting measurement precision and accuracy was approximately twice as good as required for most measurements and met requirements on all others.

Analysis of the flight data revealed that current theories need significant modification. Moreover, convection, both on Earth and in space, may have greater effect than previously believed. These findings are unexpected and immediately important to the science of Solidification and, longer term, to industrial alloy production (e.g., steel and aluminum).

Our first Journal Article was received by Physical Review on March 23 (only 6 days after Columbia landed). Subsequently, we have presented at 10 prestigious seminars and conferences including the 26th COSPAR at the World Space Conference in Hamburg, FDR. Three proceedings publications are currently in press. Moreover, as data analysis proceeds to greater depth, we are in the process of writing further papers.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	3	MS Degrees:	1
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 12/88    EXPIRATION: 12/98

PROJECT IDENTIFICATION: 963-25-05-01

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Smith, R.N., Glicksman, M.E., Koss, M.B., Bushnell, L.T., and LaCombe, J.C. "Experimental study of dendrite growth in an undercooled melt under microgravity conditions." Heat Transfer session at the 1994 ASME Winter Annual Meeting in Chicago next November (in press) 1994.

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Glicksman, M.E. "Dendritic growth in microgravity: IDGE flight 1." Aachen Foundry Institute, Aachen, FRG, 1994.

Glicksman, M.E. "Dendritic growth in microgravity: IDGE flight 1." DLR, Space Simulation Institute, Koln-Portz, FRG, 1994.

Glicksman, M.E. "The isothermal dendritic growth experiment: First results from USMP-2." Pittsburgh Chapter of the ACCG, Pittsburgh, PA, 1994.

Glicksman, M.E. "The isothermal dendritic growth experiment: First results from USMP-2." Microgravity Materials Science Conference, Von Braun Civic Center, Huntsville, AL, 1994.

Glicksman, M.E. "Space flight data from the isothermal dendritic growth experiment." At the 26th COSPAR, Hamburg, FDR, 1994.

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Glicksman, M.E. "Space flight data from the isothermal dendritic growth experiment: What have we learned?." Materials Engineering Department Seminar, Rennselaer Polytechnic Institute, Troy, NY, 1994.

Koss, M.B. "Preliminary results from the isothermal dendritic growth experiment ." At the Space Experiments Division Awards Ceremony, NASA Lewis Research Center, Cleveland, OH, 1994.

LaCombe, J.C. "Dendritic Solidification in a Microgravity Environment." Joint meeting of the Eastern New York Chapter of ASM International and the Hudson-Mohawk Section of TMS, Latham, NY, 1994.

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*Evaluation of Microstructural Development in Undercooled Alloys*

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PRINCIPAL INVESTIGATOR: Dr. Richard N. Grugel

University Space Research Association

CO-INVESTIGATORS:

W.F. Flanagan

Vanderbilt University

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**TASK OBJECTIVE:**

The objectives of this study are to be conducted in view of experiment and pertinent theory, and will, upon completion, serve to enhance our scientific understanding of solidification processes. These include evaluating the microstructural morphology, external and internal to the sample, as functions of undercooling ( $\Delta T$ ), composition ( $C_o$ ), and volume ( $V_o$ ).

**TASK DESCRIPTION:**

A series of cylindrical lead-tin alloy samples, some 16mm in length and 8mm in diameter, have been contained in quartz tubes and placed in a well controlled furnace. Nucleation of the solid is induced by one of the monitoring thermocouples when the desired undercooling, from 5 to 25K, is achieved. The sample is then metallographically prepared and microstructural development from the point of nucleation is followed. Parameters which have been varied are sample composition, degree of undercooling, and, to some extent, sample size. This experimental procedure has been paralleled by a modelling effort which predicts the microstructural development of a single dendrite that is initiated at the upper surface and grows down into the undercooled melt. Model verification has been complemented by direct observation of the solidification process using samples based on the transparent, metal analogue, succinonitrile-water system.

**TASK SIGNIFICANCE:**

The microgravity environment of space is envisioned as a novel processing arena for the solidification of metals and alloys. Contamination of high temperature and/or reactive materials is expected to be minimized as a container is not required to hold the melt and the samples are not limited in size. Greater undercoolings might also result due to elimination of heterogeneous nucleation sites with the subsequent solidification microstructure expected to consist of extremely fine constituents and/or novel phases, i.e. improved material properties. Unfortunately, processing in the microgravity environment is expensive and time consuming. Consequently, the intent of this work is to conduct a thorough, ground-based investigation which will evaluate microstructural development in bulk, undercooled alloys with the aim of ascertaining the advantage of processing in microgravity.

**PROGRESS DURING FY 1994:**

Experimentally these include: 1) developing a technique to preferentially nucleate the undercooled liquid alloy. 2) developing a computer model to follow the thermal history and microstructural evolution after nucleation. 3) successfully utilizing transparent materials to complement the metal alloys. Based on the computational and experimental results a number of conclusions can be drawn. These include: 1) Upon nucleation, the solidification velocity is initially very rapid, the dendritic and eutectic microstructure becoming finer as the undercooling is increased. However, the undercooled sample heats up very quickly and the majority of growth takes place at considerably less velocities. 2) Consequently, while the solidification microstructure is initially fine it coarsens rapidly as the velocity drops and is generally non-uniform throughout the sample. 3) The best microstructural uniformity is achieved in the sample with the least initial undercooling, i.e., 5K. Unfortunately the slow, but relatively constant, growth was interrupted by formation of equiaxed grains which effectively served to destroy the desired directionality of the sample. Through use of the organic analogue, origin of the equiaxed region observed in Pb-Sn alloys likely occurs due to fragmentation (not heterogeneous nucleation) and settling of dendritic grains which evolve at the point of nucleation. This detrimental quality would be minimized in a microgravity environment. 4) Care must be taken when measuring primary dendrite growth velocities in undercooled melts.

They do not grow at constant velocities, and what is measured may not be a primary dendrite at all but the continuation of secondary arms intersecting the visible surface. 5) While a microgravity environment may well suit to process high temperature and/or reactive "bulk" materials, rapid heating of the liquid is likely to preclude maintaining uniform, directionally fine, microstructures and /or significant amounts of an undercooled, e.g., peritectic, phase. A list of relevant publications, presentations, and an award notification follows.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/91 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-25-07-03

RESPONSIBLE CENTER: JPL

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Grugel, R.N., Fay Hua, and T.G. Wang. Evaluation of the  $\text{NaNO}_3$  -  $\text{Ba}(\text{NO}_3)_2$  eutectic system for use in investigating solidification phenomena. Journal of Materials Science Letters., (In press).

#### Proceedings

Hua, F. and R.N. Grugel. "Microstructural evolution in undercooled Pb - Sn alloys: Composition and sample size effects." Experimental Methods for Microgravity Materials Science: 6th International Symposium, 1994, TMS, eds., R.A. Schiffman and J.B. Andrews, pp. 111-115.

#### Presentations

Grugel, R.N. "Appreciating gravity during directional solidification of metals and alloys." University of Missouri-Rolla, April 20, 1994.

Grugel, R.N. and Fay Hua. "Using transparent materials to simulate solidification phenomena in metals - Microstructural development in undercooled alloys." 8th Annual Alabama Materials Research Conference, 26-27 September 1994, Tuscaloosa, AL.

Grugel, R.N. and Fay Hua. "Evaluation of microstructural development in undercooled alloys." Microgravity Materials Science Conference, June 24-25, 1994, Huntsville, AL.

Hua, F. and R.N. Grugel. "Microstructural evolution in undercooled Pb - Sn alloys: Composition and sample size effects." 123rd TMS Annual Meeting, February 27 - March 3, 1994, San Francisco, CA.

Hua, F. and R.N. Grugel. "Microstructural development in undercooled alloys." TMS Fall Meeting, October 17-21, 1993, Pittsburgh, PA.

Hua, F., R.N. Grugel, and T.G. Wang. "Analysis of microstructural development during solidification in bulk, undercooled Pb - Sn alloys." ASM-TMS Fall Meeting, October 2-6, 1994, Rosemont, IL.



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*Thermophysical Properties of Metallic Glasses and Undercooled Alloys*

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PRINCIPAL INVESTIGATOR: Dr. William L. Johnson

California Institute of Technology

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## CO-INVESTIGATORS:

D. Lee

California Institute of Technology (Caltech)

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**TASK OBJECTIVE:**

The objective is to study thermophysical properties of undercooled alloy melts and how they relate to glass formation. Toward this end, we have developed non-contact calorimetric methods to investigate the specific heat and thermal conductivity of these melts, both in the liquid and undercooled region. These quantities are essential for the development of newer, more advanced processing technologies for both existing and future materials.

**TASK DESCRIPTION:**

Non-contact AC calorimetry was successfully demonstrated on the IML-2 flight in July, 1994. We obtained information on the specific heat and thermal conductivity of liquid and undercooled Zr<sub>76</sub>Ni<sub>24</sub> and Ni<sub>60</sub>Nb<sub>40</sub> melts using TEMPUS. This data is currently being analyzed to calculate entropy and free energy functions for these melts. We will compare these quantities to their values for the corresponding equilibrium and metastable crystals to compare the relative stability of the phases. Also, we will determine the Kauzmann isentropic temperature of the alloys and compare it to the observed glass transition temperature.

In addition, the ground-based total radiance bolometer is currently being integrated onto a UHV levitation chamber for total hemispherical emissivity measurements. Measurement of temperature-dependent total hemispherical emissivity functions will allow us to unwind specific heat from undercooling data in an unambiguous manner.

**TASK SIGNIFICANCE:**

The non-contact AC calorimetry experiment is significant for many reasons. First, the thermodynamic properties of these advanced materials are a prerequisite to the development of processing technologies for them. Without knowledge of heat capacities and thermal conductivities, it is not possible to define, for example, how much power is needed to melt and cast the materials. In addition, the specific materials chosen for our experiment are the parent compounds for a new class of bulk metallic glasses that have recently been discovered by our group here at Caltech. By studying the properties of these parent compounds, we hope to better understand the bulk metallic glasses and how they form. These materials will revolutionize metallic processing technologies with their novel, superior properties. These materials can be engineered to be more ductile, slipperier, harder, lighter and more corrosion resistant than the typical materials used today. It is essential that the processing technologies for these materials be developed as quickly as possible and that, therefore, the thermophysical properties be measured.

**PROGRESS DURING FY 1994:**

The AC non-contact calorimetry method was successfully demonstrated on IML-2 in July, 1994. We were successful in obtaining data for liquid and undercooled Zr<sub>76</sub>Ni<sub>24</sub> and Ni<sub>60</sub>Nb<sub>40</sub>. This data is currently being analyzed and should provide thermodynamic data not previously measureable. Preliminary measurements indicate that the total hemispherical emissivity of an undercooled liquid changes by more than 20% between the melting temperature and the glass transition, making the standard assumption of constant total hemispherical emissivity through the undercooled region completely invalid. We will do these measurements for all samples flown on TEMPUS.

## II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 2/92    EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-25-08-07

NASA CONTRACT NO.: NAG8-954

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Lee, D., Uffelman, D., and Johnson, W. "Noncontact AC calorimetry of metals and alloys." Proceedings of NATO Workshop on Science and Technology of Rapid Solidification. West Point, NY (1994).

*Orbital Processing of High Quality Cadmium Telluride*

PRINCIPAL INVESTIGATOR: Dr. David J. Larson, Jr.

Northrop-Grumman Corporation

CO-INVESTIGATORS:

A. Levy	Grumman Research & Development Center
DiMarzio	Grumman Research & Development Center
F. Carlson	Clarkson University
J. Alexander	University of Alabama, Huntsville
D. Gillies	NASA Marshall Space Flight Center (MSFC)
J. Moosbrugger	Clarkson University

**TASK OBJECTIVE:**

The objective of this research is to investigate the influence of gravitationally-dependent phenomena (hydrostatic and buoyant) on the growth and quality of Cadmium-Zinc-Telluride (CdZnTe).

**TASK DESCRIPTION:**

Grow CdZnTe crystals in microgravity on USML-1/STS-50 using the seeded Bridgman-Stockbarger method of crystal growth. Bridgman-Stockbarger crystal growth is accomplished by establishing isothermal hot-zone and cold-zone temperatures with a uniform thermal gradient in between. The thermal gradient spans the melting point of the material (1095 °C). After sample insertion the furnace's hot and cold zones are ramped to set-point temperatures establishing a thermal gradient of 35 °C/cm and melting the bulk of the sample. The furnace is then moved further back on the sample, causing the bulk melt to come in contact with the seed crystal, thus 'seeding' the melt. The seed crystal prescribes the growth orientation of the crystal grown. Having seeded the melt, the furnace is thermally equilibrated and then the furnace translation is reversed and the sample is solidified by passing the thermal gradient down the length of the stationary sample at a uniform velocity.

**TASK SIGNIFICANCE:**

The family of II-VI compound semiconductors, of which CdZnTe is a commercially significant member, is used in the fabrication of medium and long wavelength IR sensors and beta and gamma ray nuclear detectors. Orbital processing offers the unique opportunity to significantly advance toward the goal of increased structural perfection within bulk crystals of greater chemical homogeneity. The former results from solidification and post-solidification cooling with little or no wall contact. The latter from the damping of buoyancy driven convection and concomitant growth of a homogeneous crystal under diffusion controlled growth conditions.

**PROGRESS DURING FY 1994:**

Two CdZnTe crystals were grown in the Crystal Growth Furnace (CGF) during the First United States Microgravity Laboratory Mission (USML-1). These crystals were used for comparative analysis with terrestrial baselines (CGF and commercial). The microgravity ( $\mu$ -g) and terrestrial (one-g) samples were quantitatively analyzed for chemical homogeneity, opto-electronic performance (infrared transmission), and structural perfection.

Chemical homogeneity (macrosegregation) was predicted by the process model to be low for this alloy system, even in one-g. This was confirmed experimentally. Nearly diffusion-controlled growth was achieved in the CGF-size samples (15mm OD). The effective redistribution coefficient ( $k_e$ ) of Zn in CdTe was reduced from  $k_e=1.25$  to  $k_e=1.10$  in commercial-size boules (>40mm OD), demonstrating that careful experiment control can significantly reduce macrosegregation terrestrially, even on a commercial scale. Longitudinal and radial segregation was monitored in the flight samples. It was found that the solidification record was disturbed due to unanticipated asymmetric thermal fields experienced by the flight samples. This will be corrected on the flight experiment to be conducted on USML-2.

FTIR transmission of both ground and flight materials was measured to be close to theoretical; 63% versus 66% (theoretical). This suggested that both the ground and flight materials were close to the stoichiometric composition. Infrared microscopy and x-ray measurements confirmed that the principal precipitates were Te and their size (1-10  $\mu\text{m}$ ) and density suggested that both primary flight and ground base samples experienced similar cooling rates and were close to stoichiometry.

The flight samples, however, were found to be much higher in structural perfection than the ground samples produced in the same furnace under identical growth conditions except for the gravitational level. Rocking curve widths were found to be substantially reduced, from 20/35 arc-seconds (one-g) to 9/15 arc-seconds ( $\mu\text{-g}$ ) for the best regions of the crystals. The value of 9 arc seconds equals the best reported terrestrially for this material. Morphologically, the ground samples were found to have a fully developed mosaic structure consisting of subgrains and large regions of cross-slip, whereas the flight samples exhibited discrete dislocations and no mosaic substructure or cross-slip was evident. The defect density was reduced from 50,000 -100,000 (one-g) to 500 - 500 ( $\mu\text{-g}$  sample 1) and 1200 - 3000 EPD ( $\mu\text{-g}$  sample 2). These, too, are the lowest reported.

The thermo-mechanical model suggested that the low dislocation density was due to the near-absence of hydrostatic pressure in  $\mu\text{-g}$  which allowed the melt to solidify with minimum wall content, reducing the stress transmitted to the crystal during growth and post-solidification cooling. Further, the highest quality material was predicted to be on the periphery of the boule, unlike the terrestrial samples where the best material is at the core, and this was confirmed microstructurally.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	3	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 8/90 EXPIRATION: 10/96

PROJECT IDENTIFICATION: 963-21-08-02

NASA CONTRACT NO. NAS8-38147

RESPONSIBLE CENTER: MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Carlson, F.M., and Moosbrugger, J.C. "The role of thermal stress in vertical Bridgman growth of CdZnTe crystals." MAE Department Seminar Series, Clarkson University, Potsdam, NY, September, 1994.

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Fedoseyev, A., Alexander, J.I.D., and Bessonov, O. "CNPACK - Efficient solution of large sparse linear systems." First Southeastern Linear Algebra Conference, Chattanooga, TN, May 27-28, 1994.

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Klein, J., and Larson, Jr., D.J. "Comparison of theoretical and experimental performance of a PMZF furnace for microgravity materials processing." Symposium on  $\mu$ g Solidification: Theory and Experimental Results, TMS Meeting, Pittsburg, PA, October 1993..

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*Crystal Growth of II-IV Semiconducting Alloys by Directional Solidification*

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PRINCIPAL INVESTIGATOR: Dr. Sandor L. Lehoczky

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

F. Szofran

NASA Marshall Space Flight Center (MSFC)

C. Su

NASA Marshall Space Flight Center (MSFC)

R. Scripa

University of Alabama, Birmingham (UAB)

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TASK OBJECTIVE:

The objective of this research is to investigate the effects of reduced gravity on the crystal growth of mercury zinc telluride (HgZnTe) and mercury zinc selenide (HgZnSe) alloys with respect to their compositional, metallurgical, and optical properties.

## TASK DESCRIPTION:

The investigation includes both Bridgman-Stockbarger and solvent growth methods, as well as growth in a magnetic field. The alloys are prepared by reacting pure, elemental constituents in evacuated, sealed, fused-silica ampules. The crystals are grown in a multizone furnace. The hot zone is heated above the liquidus temperature of the given alloy and the cold zone is maintained at lower temperatures to provide temperature gradient sufficient to prevent constitutional supercooling. Crystal growth is accomplished by slowly moving the ampule from the hot zone to the cold zone of the furnace. The flight portion of the investigation is being performed by using the Crystal Growth Furnace (CGF). Preparation of the samples is being done in the Space Science Laboratory of Marshall Space Flight Center. Characterization and analysis of the samples after processing is being done primarily in the same laboratory with substantial microstructural analysis being done at the University of Alabama at Birmingham. Device fabrication and characterization will be done primarily at the Rockwell International Science Center.

## TASK SIGNIFICANCE:

The anticipated results of this study will have both scientific and technological significance. The advancement in science will result from the increased understanding of the role of gravity on the fluid dynamic and compositional redistribution phenomena during the crystal growth of solid-solution semiconducting alloys having large separation between the liquidus and solidus of the constitutional phase diagrams, and from the more accurate values of material properties that can be measured using the high-quality, bulk crystals grown in space. Any advance in quality of these electronic materials has a great technological impact because of the application to infrared detectors for NASA and DOD requirements.

## PROGRESS DURING FY 1994:

A series of HgZnTe crystal ingots has been grown from pseudobinary melts by Bridgman-Stockbarger type directional solidification using the CGF Ground Control Experiment Laboratory (GCEL) furnace, as well as MSFC heat pipe furnaces. Several ZnTe crystals were also grown using a Te-solvent zone growth method. Various thermal boundary conditions and growth rates were employed and several of the ingots were rapidly quenched during the steady-state portion of growth to establish correlation between thermal conditions and melt/solid interface shapes. These experiments also indicated that the ingots can be successfully quenched and back melted to allow a rapid return to steady-state growth. The fitting of the measured crystal compositional distributions to appropriate theoretical models was used to obtain an estimate of the effective HgTe-ZnTe liquid diffusion coefficients. To assist the modeling of the pertinent heat and mass transport processes, selected portions of the pseudobinary phase diagram, thermal diffusivity and melt viscosity have been measured. Heat capacity and enthalpy of mixing for the pseudobinary melts were calculated assuming an associated solution model for the liquid. Growth experiments for

an  $\text{Hg}_{0.84}\text{Zn}_{0.16}\text{Te}$  alloy crystal were also performed in a magnetic field that showed significant fluid flow effects on the crystal compositional distributions.

A ground preprocessed and quenched sample was successfully back-melted and partially regrown in the CGF instrument during the first United States Microgravity Laboratory (USML-1) mission. The meltback interface was within 0.5 mm of the desired value. Because of the loss of power to the CGF, the experiment was terminated after approximately 39 hours into the growth period. About 5.7 mm of sample had been grown at that point. Surface photomicrographs of the recovered sample clearly showed significant topographical differences between the space- and ground-grown portions. Compositional measurements along the sample axis indicated that the desired steady-state growth for the axial composition was reached at about 3 mm into the growth. An X-ray diffraction and SEM survey of the sample showed that both the ground- and flight-portions of the ingot contained only a few grains, i.e., were nearly single crystals, and the crystallographic orientation was maintained following back-melting and space growth. The interface shape, radial compositional variations, and the quenched-in dendritic structures of the flight sample all have shown an asymmetric behavior. The compositional data strongly suggest that the most likely cause was unanticipated transverse residual accelerations.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/92    EXPIRATION: 9/96

PROJECT IDENTIFICATION: 963-21-08-04

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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*Growth of Solid Solution Single Crystals*

PRINCIPAL INVESTIGATOR: Dr. Sandor L. Lehoczky

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

F. Szofran

NASA Marshall Space Flight Center (MSFC)

D. Gillies

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The major objective of this research is to establish the limitations imposed by gravity during growth on the quality of bulk solid solution crystals having large separation between their liquidus and solidus temperatures. The important goal is to explore the possible advantages of growth in the absence of gravity.

**TASK DESCRIPTION:**

The alloy system being investigated is  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  with x-values appropriate for infrared detector applications in the 8 to 14  $\mu\text{m}$  region. Both melt and Te-solvent growth methods as well as growth in magnetic fields are being considered. The study consists of flight experimentation and ground-based experimental and theoretical work needed to establish material properties and optimum experimental parameters for the on-going flight experiment and to assist material evaluation.  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  is representative of several alloys which have electrical and optical properties that can be compositionally tuned to meet a wide range of technological applications in the areas of sensors and lasers with applications to optical computing and communications as well as the national defense. The investigation includes both Bridgman-Stockbarger and solvent growth methods, as well as growth in a magnetic field. The alloys are prepared by reacting pure, elemental constituents in evacuated, sealed, fused-silica ampules. The crystals are grown in a multizone furnace. The hot zone is heated above the liquidus temperature of the given alloy and the cold zone is maintained at lower temperatures to provide temperature gradient sufficient to prevent constitutional supercooling. Crystal growth is accomplished by slowly moving the ampule from the hot zone to the cold zone of the furnace.

The majority of the ground-based studies are being performed in Space Science Laboratory of the George C. Marshall Space Flight Center. The flight portion of the investigation is being conducted using the Advanced Automatic Directional Solidification Furnace developed by the Marshall Space Flight Center and manifested for flights on the United States Microgravity Payload series of missions. The first flight of the instrument took place in March 1994.

**TASK SIGNIFICANCE:**

The anticipated results of this study will have both scientific and technological significance. The advancement in science will result from the increased understanding of the role of gravity on the fluid dynamic and compositional redistribution phenomena during the crystal growth of solid-solution semiconducting alloys having large separation between the liquidus and solidus of the constitutional phase diagrams, and from the more accurate values of materials properties that can be measured using the high-quality, bulk crystals grown in space. Any advance in quality of these electronic materials has a great technological impact because of the application to infrared detectors for NASA and DOD requirements.

**PROGRESS DURING FY 1994:**

Over the past several years, a detailed evaluation has been performed on the effects of growth parameters on the axial and radial compositional uniformity, defect density, and optical properties in directionally solidified  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  and other similar compounds and pseudo-binary alloys. A series of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  alloy ingots ( $0 < x \leq 0.6$ ) has been grown

from pseudobinary melts by vertical Bridgman-Stockbarger type heat-pipe furnace assembly using a wide range of growth rates and thermal conditions. Several of the experiments were performed in transverse and axial magnetic fields of up to 5T. Precision measurements were performed on the ingots to establish compositional distributions and defect density distributions for the ingots. Correlation between growth rates and thermal conditions and growth interface shapes have been established for the alloy system. To assist the interpretation of the results and the selection of optimum in-flight growth parameters, the pseudobinary phase diagram ( $0 \leq x \leq 1$ ), liquid and thermal diffusivities ( $0 \leq x \leq 0.3$ ), melt viscosity, and the specific volumes as a function of temperature ( $0 \leq x \leq 0.15$ ) have been measured. From these measurements and other available data, the heat capacity, enthalpy of mixing, and the thermal conductivity of pseudobinary melts have been calculated using a regular associated solution model for the liquid phase. A one-dimensional diffusion model that treats the variation of the interface temperature, interface segregation coefficient, and growth velocity has been used to establish effective diffusion constants for the alloy system. Theoretical models have been developed for the temperature distribution and the axial and radial compositional redistribution during directional solidification of the alloys. These were used along with the experimental results to select the parameters for the first flight experiment flown on the Second United States Microgravity Payload (USMP-2) mission. A microscopic model for the calculation of point-defect energies, charge-carrier concentrations, Fermi energy, and conduction-electron mobility as functions of  $x$ , temperature, and both ionized and neutral defect densities has been developed. For selected samples, measurements were performed of electron concentration and mobility from 10-300K. The experimental data were in reasonably good agreement with theory and were successfully analyzed to obtain donor and acceptor concentrations for various processing conditions.

A five zone Bridgman-Stockbarger type furnace system designated as the "Advanced Automatic Directional Solidification Furnace (AADSf)" has been designed and developed for operating in the cargo bay of the Space Transportation System (STS). Over 15 growth experiments were performed in the development and ground-based versions of the AADSf to establish optimum operating parameters and procedure for the initial flight experiment. The experiment was successfully flown on STS62 mission as part of the Second United States Microgravity Payload (USMP-2) mission in March 1994. An approximately 15 cm long and 0.8 cm diameter  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  alloy crystal was grown at a rate of 0.78 mm/h over a period of approximately 11 days. Preliminary x-ray radiographs of the crystal indicate that the growth was successful. Characterization of the crystal is in progress.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/84 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-30-01

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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*GaAs Crystal Growth Experiment*

PRINCIPAL INVESTIGATOR: Prof. David H. Matthiesen

Case Western Reserve University

## CO-INVESTIGATORS:

Dr. J.A. Kafalas  
Dr. B.M. DitchekGTE Laboratories, Inc.  
GTE Laboratories, Inc.

## TASK OBJECTIVE:

The objective is to determine the magnitude of effects of buoyancy-driven convection on the crystal growth of bulk gallium arsenide (GaAs).

## TASK DESCRIPTION:

Selenium-doped ( $\sim 10^{-17}$ ) GaAs crystals are grown in controlled environments at selected environments affecting fluid flow as follows: (a) low-gravity (minimal convection), and (b) normal gravity in three separate orientations (vector stabilizing the temperature gradients, vector destabilizing the thermal gradient, and vector transverse to the thermal gradient), and a magnetically damped flow (the three normal-gravity orientations with either axial or radial magnetic field). The distribution of dopant is measured and compared to numerical predictions. Selected electrical and chemical properties are measured and correlated with the dopant distribution. Both macro- and micro-segregation are determined.

## TASK SIGNIFICANCE:

Gallium arsenide (GaAs), an electronic material, has two principle advantages over silicon for producing solid state electronic "chip:" its ability to emit light, useful for making lasers, and its inherently high speed, useful in high-speed computers, communication satellites, etc. To fully exploit these characteristics, the material must be of the highest quality and be uniformly doped with traces of impurities. Typically, such uniformity is determined by convection in the molten material from which GaAs crystals are grown.

This materials processing experiment, part of a larger effort to better understand and control the crystal growth process, was undertaken to investigate the effects of buoyancy-driven convection on crystal growth.

## PROGRESS DURING FY 1994:

The progress in FY1994 was very limited. This was due to the fact that the project is near completion and the balance of the work for FY1994 will be to complete for the Final Report. This is expected to be released near the end of the fiscal year.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 9/85 EXPIRATION: 4/94

PROJECT IDENTIFICATION: 963-21-05-02

RESPONSIBLE CENTER: LeRC

*Diffusion Processes in Molten Semiconductors*

PRINCIPAL INVESTIGATOR: Prof. David H. Matthiesen

Case Western Reserve University

CO-INVESTIGATORS:

Dr. W. Arnold  
Dr. A. Chait  
Dr. B. Dunbar  
Prof. D. Stroud

Case Western Reserve University  
NASA Lewis Research Center (LeRC)  
NASA Johnson Space Center (JSC)  
Ohio State University

**TASK OBJECTIVE:**

To provide purely diffusive experimental measurements of the isothermal diffusion coefficients of Ga, Sn and Sb in molten germanium with sufficient accuracy and precision to:

- a) Differentiate between model predictions of the temperature dependence.
- b) Determine the effect of dopant size and type.
- c) Determine if a "wall effect" is present.
- d) Provide input to continuum and atomistic model development.

To provide purely diffusive experimental measurements of the thermomigration diffusion coefficients of Ga, Sn and Sb in molten germanium with sufficient accuracy and precision to:

- a) Determine the effect of dopant size and type.
- b) Determine if a "wall effect" is present.
- c) Provide input to continuum and atomistic model development.

To develop a 3-dimensional, fully time dependent continuum numerical model of the germanium diffusion column, shear cell, cartridge and furnace for both earth-based and space-based experiments which accurately predicts the measured concentration profile as a function of distance in the diffusion column.

To develop atomistic models which accurately predict:

- a) The purely diffusive isothermal diffusion coefficient of a dopant in a molten semiconductor,
- b) The temperature dependency of dopants in molten semiconductors, and which:
- c) Attempts to explain the "wall effect,"
- d) Develops new empirical potentials useful for predicting other diffusion and transport properties for other molten semiconductor systems.

**TASK DESCRIPTION:**

This program of study is directed at the fundamental and applied issues pertaining to diffusion of species in the liquid state as driven by concentration gradients (Fickian diffusion) and thermal gradients (Soret diffusion). The fundamental material systems of interest for near term study are the dilute binary systems of gallium (Ga), tin (Sn) and antimony (Sb) in germanium (Ge). Systems of commercial interest for future study include the dilute binary systems of dopants in gallium arsenide (GaAs). This research program consists of three major components: an experimental measurement portion, a continuum numerical simulation portion and an atomistic numerical simulation portion.

The experimental measurement portion is designed to provide definitive measurements of the purely diffusive component of mass transfer in molten semiconductor systems. A shear cell technique will be used to directly measure the diffusion coefficients in semiconductor melts. For the Fickian diffusion case, isothermal measurements will be used to determine the diffusion coefficients. An experimental matrix will be used to determine the

dependence of the diffusion coefficients on temperature, dopant type and column diameter. For the Soret diffusion case, measurements will be made in a thermal gradient.

### TASK SIGNIFICANCE:

The fundamental mechanisms of mass diffusion in the liquid state are still unclear to the degree necessary for the prediction of diffusion of one species into another or even within itself. This observation is especially true with respect to the dependency of diffusion mechanisms on temperature and on concentration levels, as well as on the dopant type. Present estimates of diffusivity in molten semiconductors can typically provide an order of magnitude estimate only, without any information on their dependency on concentration levels and types, and on temperature and temperature gradients.

The availability of these data is of paramount importance for practical reasons as well. The relevancy of numerical modeling for the analysis and design of ground based and space experiments is directly dependent upon the accuracy of the fundamental material properties used in these simulations. These data are also important for the correct characterization and interpretation of experimental results from ground based and space experiments.

The subject of how a mass of one species diffuses through a matrix of another is, at the same time, both a very old and very new research area. That this area can encompass the small, i.e., movement of electrons in a plasma, to the very large, i.e., the depletion of the global ozone layer, merely serves to emphasize the fundamental aspects of this subject. Most manufacturing technologies at some stage, rely on diffusion processes in the solid, liquid or gas.

The need for precise measurements of the diffusion coefficients in molten semiconductors has been repeatedly pointed out. These data are required both to interpret the experimental results from previous space-based (and Earth-based) experiments and also to optimize newly envisioned experiments. Difficulties in experimental techniques and theoretical interpretations are cited for the lack of these data. This is a comprehensive program which addresses both of these issues.

### PROGRESS DURING FY 1994:

Quantified the level of mixing between adjacent shear cell segments due to the initial fluid-fluid shear.

- Numerically evaluated shearing rate.
- Numerically quantified the effect of convection on the measured diffusion coefficient.
- Established machining tolerances for shear cells.
- Numerically evaluated Soret diffusion.
- Numerically evaluated the use of an applied magnetic field to suppress convection.
- Quantified error bars to establish confidence level in the measured diffusion coefficient.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 963-21-05-04

NASA CONTRACT NO.: NCC3-293

RESPONSIBLE CENTER: LeRC

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### *The Study of Dopant Segregation Behavior During the Growth of GaAs in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. David H. Matthiesen

Case Western Reserve University

CO-INVESTIGATORS:

J. Kafalas

Viable Systems, Inc.

#### TASK OBJECTIVE:

To characterize the two selenium doped gallium arsenide crystals which were grown in the Crystal Growth Furnace (CGF) aboard the first United States Microgravity Laboratory (USML-1).

#### TASK DESCRIPTION:

During this task, the dopant distribution of the selenium in the grown gallium arsenide will be measured. Several complementary techniques will be used including optical (infrared transmission FTIR and QIR); electrical (Hall effect, C-V DLTS) and chemical (SIMS). In addition, NASA supplied acceleration measurements will be used to correlate any segregation events present in the crystal.

#### TASK SIGNIFICANCE:

By cross comparisons of the optical, electrical and chemical measurements an exacting dopant distribution will be measured in the crystal. When NASA supplies the appropriate acceleration data then, for the first time, an exacting correlation will be made between the measured segregation events and any acceleration event.

#### PROGRESS DURING FY 1994:

The segregation measurements are nearly complete. These data indicate that initially diffusion controlled growth was achieved. However, after approximately 1 cm of growth the data indicate that the segregation behavior transitioned to that of complete mixing. The acceleration data is being examined for any causes.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 5/92 EXPIRATION: 10/96

PROJECT IDENTIFICATION: 963-21-05-01

NASA CONTRACT No. NAS8-39722

RESPONSIBLE CENTER: MSFC

#### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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*Temperature Dependence of Diffusivities in Liquid Metals*

PRINCIPAL INVESTIGATOR: Prof. Franz E. Rosenberger

University of Alabama, Huntsville

CO-INVESTIGATORS:

R. M. Banish

University of Alabama, Huntsville

**TASK OBJECTIVE:**

This research aims at advancing the understanding of diffusion mechanisms in liquid metals and alloys through accurate diffusivity measurements over a wide range of temperatures, including the proximity of the materials melting points.

**TASK DESCRIPTION:**

Toward the above objective we are pursuing the following tasks:

- development of an efficient technique for dynamic in-situ measurements of diffusivities in melts as a function of temperature;
- development of a flight-certified hardware package to automatically perform such diffusivity measurements under reduced gravity conditions and on Earth;
- investigation of the significance of the “wall effect” in diffusion capillaries;
- exploration of convective contamination of the diffusivity measurements on Earth through numerical modelling;
- exploration of the possibility to simulate low gravity diffusion conditions in conducting liquids on Earth through the application of magnetic fields;
- measurements of diffusivities of selected materials that will be chosen according to class-like molecular interaction behavior in the liquid.

Diffusivities will be determined from temporal records of evolving concentration profiles through multi-detector measurements of radioactive tracer emission. An initially solid, cylindrical sample contains a radioactive isotope at one end. After melting, radiation escaping through small bores in an isothermal liner/radiation shield is monitored via a chain of detectors. Data evaluation is facilitated by a novel algorithm, which is not limited to the simple initial conditions traditionally used in diffusivity studies. The algorithm permits data deduction from any sequence of concentration distributions. Hence, diffusivity data can be gathered over a range of temperatures in a single experiment. Utilizing the different radiation absorption behavior of different photon energies, we will investigate the significance of the “wall” effect. This effect is currently believed to contaminate diffusion studies in narrow capillaries used to suppress convection at normal gravity.

**TASK SIGNIFICANCE:**

The diffusion of species in melts and its temperature dependence is important for the product quality in numerous metallurgical and semiconductor manufacturing processes. Hence, a detailed understanding of diffusion in liquid metals and alloys is essential for an efficient improvement of numerous technological processes. However, the accurate measurement of diffusivities in liquids is hampered by difficult-to-control transport contributions from convection. At this point, due to the complex structure of liquids, our theoretical understanding of diffusion in liquids is limited. Theoretical models of diffusion in liquids abound. Yet, a verification of these models requires more accurate diffusivity data for wider temperature ranges than are currently available. This investigation will provide accurate data for selected materials and, hence, will further both process development as well as fundamental science.

### PROGRESS DURING FY 1994:

A novel algorithm was developed for the evaluation of diffusive concentration profiles without use of boundary conditions and specialized initial conditions.

A conceptual design was completed based on detailed sensitivity and dimensional analyses of the optimal diffusion capillary and collimator geometry considering experiment time, spatial resolution and diffusivity data precision, detector sensitivity and minimization of the radioactive dose, isotope half-life and collimator/radiation shield absorption characteristics.

An isothermal diffusion oven with minimal power consumption, adequate radioactive shielding and permitting ready sample exchange, was designed and built.

A boron nitride coated graphite heater cartridge was selected for its uniform radioactive absorption characteristics and tested under oxidizing conditions over the temperature range to be used in the experiments.

Following extensive testing of various detectors for energy resolution at the elevated operation temperatures to be expected in a flight experiment, CdZnTe detectors were chosen and integrated with miniturized data acquisition electronics.

The development of software for data acquisition and evaluation was started.

Sample preparation and ampoule filling procedures were tested with particular concern for obtaining non-wetting conditions between the metal and ampoule material.

A design verification and sensitivity testing procedure for the whole experimental setup was developed utilizing a solid, radioactive sample to be mechanically moved along the diffusion direction in liquid samples.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 2/93 EXPIRATION: 8/94

PROJECT IDENTIFICATION: 963-25-08-10

NASA CONTRACT NO. NAS8-39718

RESPONSIBLE CENTER: MSFC



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*Double Diffusive Convection during Growth of Lead Bromide Crystals*

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PRINCIPAL INVESTIGATOR: Dr. N. B. Singh

Westinghouse Electric Corporation

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The main objective of this program is to evaluate, understand and eliminate thermosolutal convection during the crystal growth of  $\text{PbBr}_2$ -AgBr systems. The program will provide a quantitative understanding of convective effects and a correlation of experimental data with theories developed for thermosolutal convection will be carried out. For the  $\text{PbBr}_2$ -AgBr system less dense solute causes the convective (thermosolutal) instability in addition to morphological instability. Also, this system is optically transparent and we can monitor the interface shape to study the convective and morphological instabilities. The technical objectives of this program are to define the parameter at normal gravity to minimize the thermosolutal convection during growth of doped lead bromide crystals to achieve homogeneous distribution of dopant, significantly reduce the optical and acoustic scattering caused by convection during lead bromide crystal growth, and produce lead bromide crystals with unparalleled optical homogeneity for advanced device applications. This will be achieved by experimentally verified stability diagrams and direct observations on solid-liquid interface during crystal growth.

**TASK DESCRIPTION:**

To achieve these objectives, crystal growth experiments will be conducted on earth and in space. Measurements involving Rayleigh number as a function of aspect ratio, and the radius of the growth tube to the length of the melt column, will be made. Experimental results will be compared with the stability diagram to test the validity of morphological and convective stability theories.

**TASK SIGNIFICANCE:**

The scientific objectives of this program are to understand the thermosolutal convection during the crystal growth of  $\text{PbBr}_2$ -AgBr system. This will be achieved by growing five crystals at five different concentrations, which will lead to different solutal convective levels. The experimental values of the concentration distribution will be compared with the theories based on pure diffusional growth to evaluate the effect of convection. Also, numerical studies will be carried out to study the convective and morphological instabilities, and to determine the critical concentration of dopant for a particular growth velocity and gravity level. Theoretical instability diagrams will be compared with the experimental studies. Relevant analytical characterization techniques are to be used to evaluate the effect of convection on crystal quality. These studies will provide basic data on convective behavior in doped lead bromide crystals grown by the commercially important Bridgman process.

**PROGRESS DURING FY 1994:**

Minutes of the March 1992 Flight Science Readiness Review have been published. Dr. Singh is taking action based on the recommendations of the review.

To study the effect of thermal convection, four lead bromide crystals were grown at different thermal Rayleigh numbers. X-ray rocking curves, X-ray contour scans and chemical etchpit studies showed that crystal quality decreased with increasing thermal Rayleigh number. The crystal grown at lowest thermal Rayleigh number showed highest quality and the crystal grown at highest thermal Rayleigh number showed the worst quality.

Lead bromide-silver bromide crystals were grown in a Bridgman furnace at a thermal gradient of 21 K/cm and pull rate of  $2.3 \times 10^{-5}$  cm/s. The measured concentration of silver did not match with the values predicted by theories based on pure diffusive or convective transport. X-ray rocking curves and contour scans for (010) plane showed that

crystal grown at lower dopant concentration had better quality. The crystals grown at lower solutal Rayleigh number exhibited small variation of lattice parameters and better homogeneity in dopant distribution. Results of etchpit studies also supported these results.

We have carried out a crystal growth run at a temperature gradient of 20 K/cm, to test the validity of stability theory. We are using a charge containing 5000 ppm silver bromide. When we have steady-state conditions, the interface is flat. When we start pulling the growth tube, up to the pull rate of 0.23 micrometer per second, we did not observe any significant change in the shape. At the pull rate of 0.29 or higher, we observed that the interface turns concave, followed by a depression in the middle. We observed very interesting phenomena with the increasing time. We observed that the shape of the interface is changed significantly and can be explained on the basis of toroidal flow, just as theory had predicted. A cellular or dendritic microstructure later developed within this depressed interface region. When we stopped the pulling of the growth tube, the interface recovered its original flat shape. We did not observe this phenomena in the pure lead bromide system and identical crystal growth conditions. We are excited to have experimentally confirmed the predicted region of the interface instability under convective growth conditions. This type of instability can be explained on the basis of toroidal fluid flow. This type of flow will not occur in the pure system and the pressed pit at the interface occurs under the point where the flow field converges. These results are further justification for studying this system in low gravity. We are planning to start next run containing 500 ppm and we hope to increase the growth velocity 10 times to observe the instability.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/93 EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-24-05-01

RESPONSIBLE CENTER: LeRC

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*Particle Engulfment and Pushing by Solidifying Interfaces*

PRINCIPAL INVESTIGATOR: Prof. Doru M. Stefanescu

University of Alabama, Tuscaloosa

CO-INVESTIGATORS:

P. Curreri

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The primary objective of this task is to further develop the existing understanding on pushing and engulfment of particles by planar liquid/solid interfaces during solidification of metallic alloys.

The approach towards achieving this objective is by developing an analytical and then a numerical model describing this phenomenon, with a parallel experimental validation effort. The numerical model is required in order to include the effect of convection in the liquid on interface morphology and the particle/interface interaction. The interaction between an insoluble ceramic particle and a liquid-solid interface during solidification is significantly influenced by gravitational acceleration. Since the interaction between an insoluble ceramic particle and a liquid-solid interface during solidification is significantly influenced by gravitational acceleration, microgravity experiments will be performed to validate these models and to further contribute to the experimental data base for metals/ceramic particulate mixtures.

**TASK DESCRIPTION:**

To acquire further insight in the physics of particle behavior, directional solidification experiments with transparent organic materials doped with polystyrene particles were scheduled. It was planned to use results from these experiments to validate the analytical model developed so far.

Ground based experiments under controlled solidification conditions with Al-SiC systems to document pushing/engulfment phenomena in metallic systems were also scheduled. In addition several tasks required in the flight requirements definition effort were to be performed.

**TASK SIGNIFICANCE:**

It is of fundamental and practical importance to understand and control particle behavior at the solid-liquid interface during solidification. Particle behavior determines the uniformity of their distribution in the matrix. Uniformity of particle distribution is of great significance as it dictates the mechanical and physical properties of the composites.

Introduction of insoluble ceramic particles in a metal matrix primarily involves three stages: transfer of particles from gas to liquid, interaction of particles in the liquid state and finally transfer of particles from liquid to solid. It is the last stage that is the most important and yet the least understood since it is the outcome of the interaction of numerous solidification variables. The anticipated results from this research program will provide a much better understanding of this stage of metal matrix composite processing and therein lies its significance.

**PROGRESS DURING FY 1994:****Modeling:**

Through a combined thermal and force field calculation a model has been developed to predict particle behavior at the interface. The derived equations predict the formation of bumps or troughs on the melt interface behind the particles. This was confirmed by experiments on transparent systems containing SiC, polystyrene, or gas bubbles. The model allows calculation of the critical velocity for particle engulfment:

$$V_c = A(G/C_o)^{1/2} R^{-1}$$

where,  $C_o$  is the equilibrium solute concentration,  $G$  is the thermal gradient ahead of the interface,  $R$  is the radius of

curvature, and A is a constant that includes material parameters. Experimental and analytical modeling work on transparent systems have demonstrated that in the presence of convective fluid flow the critical velocity for particle engulfment increases. This is primarily due to particles moving away from the interface with a velocity comparable to the fluid flow velocity.

### Progress in Experimental Work:

Transparent organic materials doped with polystyrene particles have been directionally solidified. In-situ behavior of these particles was observed under variable solidification conditions. Results from these experiments were used to validate the analytical model developed so far. A better understanding of the role of interface morphology, e.g., planar or columnar, was also achieved.

Metallic systems are opaque to light microscopy. Determining the position of the particles before and after directional solidification is important to accurately determine the critical velocity for particle engulfment. Thus it was necessary to evaluate several non destructive methods. Samples have been sent out to three different organizations (Panametrics, Boeing, and Argonne National Laboratory) for initial evaluation.

Water spray quenching experiments in the directional solidification furnace have been performed on both alumina and graphite crucibles and the corresponding cooling rates have been determined. This work was necessary for the definition of flight requirements.

A cartridge design and a method to fabricate flight samples incorporating three thermocouple tips to monitor thermal profile during the experiments has been developed. These samples will be tested at CNES.

The critical velocity for maintaining a planar interface in 99.999% Al has been determined to be 60  $\mu\text{m/s}$ .

Since spherical SiC particles required for the experiments were not available, a sputtering technique was used to coat spherical Ni particles with SiC. Spherical particles processed in this way have been characterized for coating uniformity and thickness.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 963-25-08-11

NASA CONTRACT NO. NAS8-38715

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Ahuja, S., Stefanescu, D.M., and Dhindaw, B.K. "Role of processing/system parameters on the interaction between liquid/solid interfaces and insoluble particles." Cast Metal Matrix Composites, Proceedings of the 2nd International Conference, D.M. Stefanescu and S. Sen, eds., AFS, Des Plaines, IL, 44-56 (1993).

Pang, H.T., Stefanescu, D.M., and Dhindaw, B.K. "Influence of interface morphology on the pushing/engulfment transition of polystyrene particles in succinonitrile + water matrices." Cast Metal Matrix Composites, Proceedings of the 2nd International Conference, D.M. Stefanescu and S. Sen, eds., AFS, Des Plaines, IL, 57-69 (1993).

#### Books

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*Crystal Growth of ZnSe and Related Ternary Compound Semiconductors by Physical Vapor Transport*

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PRINCIPAL INVESTIGATOR: Dr. Ching-Hua Su

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

R. Brebrick

Marquette University

M. Volz

NASA Marshall Space Flight Center (MSFC)

Y. Sha

Universities Space Research Association (USRA)

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NASA Marshall Space Flight Center (MSFC)

S. Sanghanitra

Santa Barbara Research Center

S. Johnson

Santa Barbara Research Center

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**TASK OBJECTIVE:**

The investigation consists of extensive ground-based experimental and theoretical research efforts and concurrent flight experimentation. The objectives of the ground-based studies are to obtain the experimental data and conduct the analyses required to define the optimum growth parameters for the flight experiments, perfect various characterization techniques to establish the standard procedure for material characterization and quantitatively establish the characteristics of the crystals grown on Earth as a basis for subsequent comparative evaluations of the crystals grown in a low-gravity environment, and develop theoretical and analytical methods required for such evaluations.

**TASK DESCRIPTION:**

The crystal growth experiment will use a novel vapor transport three-thermal-zone heater translating method. The Crystal Growth Furnace (CGF) or Advanced Automated Directional Solidification Furnace (AADSf) will be ideal for this experiment because they provide two high-temperature end zones and a booster heater at the center of the furnace with translation capability. Using this technique, large single crystals of CdS, CdTe, PbSe, and ZnTe have been grown successfully in this laboratory.

**TASK SIGNIFICANCE:**

The materials to be investigated are ZnSe and related ternary semiconducting alloys, e.g.,  $\text{ZnS}_x\text{Se}_{1-x}$ ,  $\text{ZnSe}_{1-x}\text{Te}_x$ , and  $\text{Zn}_{1-x}\text{Cd}_x\text{Se}$ . These materials are useful for opto-electronic applications such as high efficient light emitting diodes and low power threshold and high temperature lasers in the blue-green region of the visible spectrum. The recent demonstration of its optical bistable properties also makes ZnSe a possible candidate material for digital optical computers. Compositional non-uniformity, microstructural crystal defects (e.g., dislocations, small-angle grain boundaries, and second phase precipitates), and deviation from stoichiometry can seriously limit state-of-the-art device performance and future device applications. The reduction of gravity-driven convective fluid flows in a low-gravity environment is expected to be advantageous in minimizing these compositional variations and structural defects.

**PROGRESS DURING FY 1994:**

1. Fundamental of transport theory was confirmed by simultaneous measurements of partial pressures and transport rate.
2. Growth parameters for horizontal unseeded growth of ZnSe were optimized.
3. Characterization methods to assess the effect of gravity on the grown crystals were established.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 963-21-08-09

NASA CONTRACT NO. NAS8-39718

RESPONSIBLE CENTER: MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

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*Measurement of Viscosity and Surface Tension of Undercooled Melts*

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PRINCIPAL INVESTIGATOR: Dr. Julian Szekely

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this investigation is to utilize the electromagnetic levitation unit, TEMPUS, on IML-2 to measure the viscosity and surface tension of undercooled metallic melts. To date, little study has been made of the thermophysical properties of undercooled melts, and a controversy exists over whether the temperature dependence of the viscosity obeys an Arrhenium-type or a power-law relationship.

**TASK DESCRIPTION:**

In this investigation, a "squeezing" force will be applied to a suitably-positioned sample to induce oscillations. The rate of decay of the amplitude of these oscillations will be observed in order to measure the viscosity at a number of temperatures in the undercooled regime, while the frequencies of the oscillation modes will be used to deduce the surface tension at these temperatures.

Our effort consists largely of a comprehensive program of mathematical modeling designed to give a detailed understanding of what can be expected from the flight experiment. To date, the main thrust of the modeling work has been to develop the methodology and to perform calculations predicting the behavior of levitation-melted/electromagnetically-positioned metallic droplets under both Earthbound and microgravity conditions.

**TASK SIGNIFICANCE:**

The main purpose of the work was to be able to predict the electromagnetic forces and heating rates, electromagnetically-driven velocity fields within the sample, the transient behavior of the system, and the deformation of the sample. The accuracy of the computational models has been checked by comparison with available analytical results and the results of ground-based experiments.

**PROGRESS DURING FY 1994:**

During the IML-2 mission this experiment was performed in the TEMPUS facility. The surface tension and viscosity of undercooled metallic melts - including both pure metals (Cu, Au, and Ni) and alloys (AuCu) - were measured. The oscillating drop technique with electromagnetic levitation was used to measure the thermophysical properties in a containerless fashion. By this method, a metallic sample was positioned, melted, and squeezed by high-frequency electromagnetic fields in TEMPUS. Once the sample is molten, it is squeezed and deformed by a brief current about its equilibrium free surface shape. The surface tension can be related to the frequency of the oscillations, and the viscosity can be derived from the rate at which the oscillations are damped. The experiments were performed in microgravity in order to eliminate the strong electromagnetic forces that are required to levitate metallic samples on Earth. These strong forces drive turbulent flow inside the sample, making it impossible to measure viscosity and deforming the sample to an extent that makes measurement of the surface tension more difficult.

For three experiments mentioned above (Au, AuCu, and ZrNi), the digital data contained in the rau-telegram were analyzed with respect to the heater and positioner control voltages and temperature. The temperature data were rescaled using the effective emissivities determined at the melting plateaus, which occurred at known temperatures. Surface tension values for Au, AuCu, and ZrNi have been determined, and the results are very promising and represent the first surface tension data obtained in microgravity.

## II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 12/90 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-25-08-08

NASA CONTRACT No.: NAG8-970

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

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Schwartz, E. and Szekely, J. "The shape of liquid metal droplets in electromagnetic levitation experiments considering internal fluid flow." Proceedings of Experimental Methods for Microgravity Materials Science, 1994 TMS Annual Meeting, San Francisco, CA, 1994, 73-79.



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*Test of Magnetic Damping of Convective Flows in Microgravity*

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PRINCIPAL INVESTIGATOR: Dr. Frank R. Szofran

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

S. D. Cobb

NASA Marshall Space Flight Center (MSFC)

M. B. Robinson

NASA Marshall Space Flight Center (MSFC)

M. P. Volz

NASA Marshall Space Flight Center (MSFC)

S. Motakef

Computer Assisted Process Engineering (CAPE)

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**TASK OBJECTIVE:**

The objectives of this study are: to test experimentally the validity of the modeling predictions applicable to the magnetic damping of convective flows in conductive melts as this applies to the directional solidification of semiconductor and metallic materials in the reduced gravity levels available in low Earth orbit; and to assess the effectiveness of magnetic fields in reducing the fluid flows occurring in these materials during space processing that result from density gradients (driven by the residual steady-state acceleration or g-jitter) or surface tension gradients (Marangoni flow). To achieve these fundamental objectives, the following specific objectives will be pursued:

- To determine the relative effectiveness of transverse and axial magnetic fields in suppressing convective flows in 1g driven by gravity, vibration, or surface tension gradients;
- To test the validity of magnetohydrodynamic modeling predictions in characterizing the effectiveness of an axial magnetic field for suppressing convective flows in 1g.

**TASK DESCRIPTION:**

To achieve the objectives of this investigation, we will carry out a comprehensive ground-based program using a carefully chosen set of materials. Some of these materials have been intensely studied in environments that have not simultaneously included both low gravity and an applied magnetic field. These include a dilute alloy (Ga-doped Ge) in which solutal effects will be negligible and three solid solutions—Ge-Si, InSb-GaSb, and Cu-Ni—with liquid density ratios of 2.18, 1.07, and 1.012, respectively. Thus, during Bridgman-Stockbarger solidification with the solid on the bottom, Ge-Si has a strongly stabilizing solutal density variation, InGaSb is very mildly stabilizing with previous results showing substantial mixing, and Cu-Ni is even less stabilizing. All four systems will be processed by the Bridgman method using two diameters. In addition, the Ga-doped Ge and Ge-Si systems will be float-zoned to study the effects of magnetic suppression of Marangoni convection.

**TASK SIGNIFICANCE:**

During directional solidification of semiconductors, generation of destabilizing temperature gradients in the melt is unavoidable, resulting in buoyancy-induced convective mixing of the liquid phase. On Earth this convective mixing is generally very intensive and interferes with segregation of melt constituents at the growth front leading to less than optimum quality crystals. Crystal growth in space provides the opportunity to reduce the convective intensity and, for some classes of systems and charge sizes, achieve mass transfer diffusion-controlled growth. Magnetic damping of convection in electrically-conductive melts, however, can be used to provide a higher degree of control on convection in the melt. Thus our understanding of convective influences can be further advanced, and our ability to interpret space experimental results may be significantly improved.

**PROGRESS DURING FY 1994:**

During FY94 a new thermal control system was installed to provide excellent temperature control at all fields up to 5 T and samples of Ge:Ga, InGaSb, and GeSi were grown and analyzed. Emphasis was on the Ge:Ga system.

The initial thermal control system used time proportioning and ac current and, while adequate for zero field work, was not satisfactory for use in high magnetic fields. The current system uses dc current that is slowly varied but not turned off and on to achieve temperature control. Temperature control of better than  $\pm 0.5$  °C at all fields is routine.

The significant accomplishments of the project include:

- Demonstrated mechanical interface demarcation in Ge:Ga at fields up to 5 T;
- Reproducibly observed diffusion limited growth at 5 T in Ge:Ga;
- Developed a furnace-specific model for Ge:Ga growth which correctly predicted the field needed to achieve diffusion-controlled growth;
- Demonstrated the growth of single-crystal Ge:Ga at 5 T;
- Observed major difference in growth characteristics in  $\text{Ge}_{0.95}\text{Si}_{0.05}$  between 0 and 5 T;
- Observed significant departure from complete mixing behavior in  $\text{In}_{0.2}\text{Ga}_{0.8}\text{Sb}$

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-21-08-10

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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*Vapor Growth of Alloy-Type Semiconductor Crystals*

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PRINCIPAL INVESTIGATOR: Dr. Heribert Wiedemeier

Rensselaer Polytechnic Institute

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objectives of this research are: the establishment of experimental trends for the relation between convective flow, mass flux, and crystal morphology; and the identification of microgravity effects and crystal properties for the ternary semiconductor mercury cadmium telluride (HgCdTe).

For this purpose, thin epitaxial layers of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  will be grown on (100)CdTe substrates during the USML-2 mission to observe the effects of microgravity on the morphology of the substrate-layer interface and of the epitaxial layer.

**TASK DESCRIPTION:**

This experiment requires the hot zone to be 625 °C and the cold zone to be 455 °C. The total duration of the experiment is 16 hours. The ampoule assembly is designed to be 160 mm in length, 18 mm outer diameter, and about 31 grams total weight. A cadmium telluride single crystal and a sapphire disc are used for the epitaxial crystal growth as substrate and substrate support, respectively. Four time intervals are required for crystal growth, namely, heat-up, annealing, growth, and cool-down periods.

Identical experiments, except for the level of gravity, are performed on ground and in space to provide a direct comparison of results.

**TASK SIGNIFICANCE:**

The lateral and axial compositional homogeneity (distribution) of the major and dopant components is expected to be more uniform for the space-grown epitaxial layers. The density of dislocations, of strain-induced defects, and possibly the number of inclusions are expected to be considerably reduced relative to ground-control specimens.

In addition, observations of the effects of reduced gravity on the formation of defects at the growth interface and on the propagation of these "birth defects" into the layer are of basic scientific and technological significance.

**PROGRESS DURING FY 1994:**

On-going ground-based experiments in the P.I.'s laboratory and in the GCEL furnace revealed the effects of growth temperature on the critical transient time of layer growth. These experiments provide the basis for the definition and refinement of flight experiment parameters.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 3

TASK INITIATION: 10/90 EXPIRATION: 10/96

PROJECT IDENTIFICATION: 963-21-08-03

NASA CONTRACT NO. NAS8-39723

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

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#### Presentations

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Superfluid Transition of  $^4\text{He}$  in the Presence of a Heat Current

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PRINCIPAL INVESTIGATOR: Prof. Guenter Ahlers

University of California, Santa Barbara

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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## TASK OBJECTIVE:

The objective of this project is to study the superfluid transition in a heat current. One issue which we are addressing is whether the superfluid transition remains continuous in the presence of a heat current. A second objective is to make measurements of the effective conductivity of the system very close to but slightly above the transition temperature  $T_\lambda$  as a function of the current.

## TASK DESCRIPTION:

Theoretical work by Onuki has predicted that the transition will be hysteretic. We are looking for this hysteresis in a finite current. Onuki's theory does not take the effect of gravity into consideration, and it is not clear to what extent the gravitationally induced inhomogeneity will hide the predicted effect. Recent calculations by Haussmann and Dohm have indicated that a nonlinear range of parameter space should be accessible where the conductivity will depend upon the current. This range will be exceedingly close to  $T_\lambda$  where the ultra-high resolution thermometry developed previously in our laboratory will be essential, and where gravity effects will play an important role.

## TASK SIGNIFICANCE:

We expect that our earth-bound measurements will yield information about possible advantages to be gained from micro-gravity experiments. We will have to determine whether gravity effects completely obscure the nonlinear regime, thus necessitating microgravity experiments in order to make these nonlinear effects observable, or whether useful information can be obtained in an earth-bound laboratory.

## PROGRESS DURING FY 1994:

Since January 1, 1994 we have continued to study the finite heat current effect on the thermal conductivity of  $^4\text{He}$  near the lambda transition. The following progress has been made:

We finished developing a computer code to calculate the two-dimensional temperature field in our cell. The simulation gave results in agreement with known experimental results and enhanced our understanding of our experimental data, particularly in the HeI--HeII coexistence region. Furthermore, it indicates that the side wall of our cell has a negligible effect in studying the nonlinear thermal conductivity of  $^4\text{He}$ .

To analyze the experimental data we obtained last year, we have developed a computer code to do nonlinear least squares fitting of our experimental data. This is nontrivial due to the complexity of the model which involves a number of numerical procedures. With this program we have performed extensive data analysis using different trial models of conductivity.

Quantitative comparison of our data with the theoretical prediction by Haussmann and Dohm requires the numerical evaluation of the Haussmann and Dohm formulas. With generous support from Dr. Haussmann and Professor Dohm we have developed a computer program to evaluate the theoretical predictions for the thermal conductivity of  $^4\text{He}$  as a function of heat current and distance from  $T_\lambda$ . This enabled us to make quantitative comparison between our experimental data and the Haussmann and Dohm theory. We shared our code with Dr. Rob Duncan's group at Sandia National Lab, since it is equally important to their NASA sponsored research.

Our initial data analysis indicated that further experimental data with cells of different thicknesses are needed. We constructed an experimental cell with 0.4mm thickness, as compared to 1.1mm in the previous experiment. This cell was installed in our apparatus, cooled down, and used to take data. The analysis of this second set of results is now well under way. A third cell, of thickness 0.8 mm, will also be used in the near future.

Finally, a detailed written report was completed and submitted to JPL following the January 1994 NASA workshop in Washington DC where we (Professor Guenter Ahlers and Dr. Feng-Chuan Liu) each presented an oral report.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-17

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Ahlers, G. "Dynamics of the superfluid transition of  $^4\text{He}$ ." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

Liu, F.C., and Ahlers, G. "The effect of a finite heat current on the lambda-transition and on the thermal conductivity of  $^4\text{He}$ ." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

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Microgravity Test of Universality and Scaling Predictions Near the  $^3\text{He}$  Critical Point

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PRINCIPAL INVESTIGATOR: Dr. Martin B. Barmatz

Jet Propulsion Laboratory (JPL)

## Co-INVESTIGATORS:

U.E. Israelsson

Jet Propulsion Laboratory (JPL)

J. Rudnick

University of California, Los Angeles

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## TASK OBJECTIVE:

The objective of this task is to test the universality and scaling laws at the liquid-gas critical point of  $^3\text{He}$  in a microgravity environment. The task objectives will include 1) precision measurements of the isothermal compressibility along the critical isochore to determine the critical exponent  $\gamma$  and 2) precision measurements of the constant volume specific heat along the critical isochore to determine the critical exponent  $\alpha$ .

## TASK DESCRIPTION:

Theories describing the behavior of thermophysical properties near critical points were developed using the concept of scaling laws. These models led to the definition of universality classes where critical points of the same class are predicted to have the same critical exponents. Efforts to validate the scaling law predictions near a liquid-gas critical point in ground-based laboratories are limited due to the gravity induced vertical density gradient associated with the divergence of the isothermal compressibility. This density gradient becomes appreciable as the critical point is approached leading to a significant smearing of the transition. Calculations have shown that in a microgravity environment ( $10^{-6}g$ ) accurate specific heat and isothermal compressibility measurements could be obtained two orders of magnitude in reduced temperature closer to the critical point. Techniques are being developed for the simultaneous measurement of both static (specific heat, sound velocity, and compressibility) and dynamic (sound attenuation and dispersion) properties. These studies will require accurate measurements of pressure ( $Dp/p \sim 10^{-11}$ ), density ( $D\rho/\rho \sim 10^{-9}$ ), and temperature ( $DT/T \sim 10^{-9}$ ).

## TASK SIGNIFICANCE:

The ability to perform these simultaneous measurements in microgravity should provide a very stringent test of the universality predictions.

## PROGRESS DURING FY 1994:

During FY94, we designed, fabricated, and tested a  $\text{GdCl}_3$  high resolution thermometer for operation at the  $\text{He}^3$  critical point,  $T_c=3.3\text{K}$ . This  $\text{GdCl}_3$  HRT was shown to have the same sensitivity at the  $\text{He}^3$  critical point as a CAB HRT has at the Lambda Point. A new magnetostrictive fluid transfer valve was also demonstrated at liquid helium temperatures for use at the  $\text{He}^3$  critical point.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-04-07

RESPONSIBLE CENTER: JPL

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Proceedings**

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Hahn, I., and Barmatz, M. "Application of  $\text{GdCl}_3$  thermometry at temperatures near the liquid gas critical point of  $^3\text{He}$ ." Proceedings of the NASA JPL 1994 Microgravity Low Temperature Physics Workshop.

Rudnick, J., and Barmatz, M. "Effects of random motions on critical point measurements." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.



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*Measurement of the Heat Capacity of Superfluid Helium in a Persistent-Current State*

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PRINCIPAL INVESTIGATOR: Dr. Talso C. Chui

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of the task is to detect any changes in the heat capacity of helium as result of superfluid flow very near the superfluid transition temperature.

**TASK DESCRIPTION:**

The flow will be created in a toroidal shaped calorimeter in the form of persistent current. The heat capacity is then measured from below to above the transition, where the persistent current will decay to zero. The heat capacity will subsequently be remeasured below the transition to detect any difference. If the experiment shows that the heat capacity is different with superfluid flow, then a space experiment can be designed to map out the heat capacity curves as a function of temperature and superfluid velocity.

**TASK SIGNIFICANCE:**

The results will be compared to the dynamic renormalization group theory, which have recently been applied to calculate the expected results. The theory, which involves three adjustable parameters, is remarkably successful in explaining the thermal conductivity and the second sound damping near the lambda transition. The proposed experiment will give a much more stringent test of the theory because this new experimental situation allows the theory to make precise predictions without any additional adjustable parameters.

**PROGRESS DURING FY 1994:**

Recently there was new experimental evidence supporting the idea initially proposed by us that in heat flow experiment near the lambda transition, the first rise in temperature is associated with vortex creating of the type proposed by Langer and Fisher to explain the intrinsic critical velocity observed by Clow and Reppy in a superfluid gyroscope. If this interpretation is correct, it would affect the experiment design significantly. First, the real shift in the lambda transition temperature as predicted by the renormalization group theory (RG) cannot be easily reached. Second, the region where vortex creation is not important, and the RG prediction can be realized may be at  $|t| < 10^{-4}$  where  $t$  is the reduced temperature. Therefore in a heat current arrangement, measurement of the heat capacity at  $|t| > 10^{-4}$  would probe the effect of vorticities on the heat capacity rather than the effect of RG.

Based on these development, the emphasis of the experiment has shifted to charting the region of experimental interest where the RG prediction can be realized. And then designing an experiment that would work in this region. An experimental cell has been fabricated for this purpose. The apparatus has been in operation for over a year with high resolution thermometers capable of resolving temperature to  $5 \times 10^{-11}$  K/Hz<sup>1/2</sup>. The germanium resistance thermometers and the high resolution thermometers are fully interfaced to a computer with real time graphic software based on LabView G language. The PID temperature controllers are based on software digital control implemented with Labview. We are current working at integrating the cell and all the software and hardware together. A first run is planed early next year.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-04-08

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Chui, T.C.P., Day, P., Hahn, I., Nash, A.E., Swanson, D.R., Nissen, J.A., Williamson, P.R., and Lipa, J.A. "High resolution thermometers for ground and space applications." Proceedings of the 15th International Cryogenic Engineering Conference, Genova Italy 1994.

Day, P., Chui, T., and Israelsson, U. "Heat capacity of helium under superfluid flow conditions." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

Tonucci, R.J., Chui, T.C.P., Israelsson, J.A. and Lipa, J.A.. "Application of nanochannel array for the studies of finite size effects near a second order phase transition." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

Yeh, N.C., Strayer, D.M., Chui, T.C.P., and Lysek, M.J. "Exploring the helium lambda transition using a high-Q microwave cavity." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

Nonequilibrium Phenomena Near the Lambda Transition of  $^4\text{He}$

PRINCIPAL INVESTIGATOR: Dr. Talso C. Chui

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

Dr. Ulf Israelsson

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

The objective of this project is to obtain information on the order-parameter relaxation time in superfluid helium near the lambda transition.

**TASK DESCRIPTION:**

The experiment will measure the heat capacity of superfluid helium near the lambda transition under a pressure oscillation at variable frequency. There will be a small change in the averaged heat capacity when the frequency is increased above the reciprocal of the order-parameter relaxation time, thus allowing a measurement of the relaxation time. The proposed technique is well suited for performance in space where close approach to the transition is possible without the smearing effect of gravity induced inhomogeneity. Since the relaxation time is predicted to slow down considerable near the transition, getting closer to the transition will avoid problems on earth associated with extremely fast relaxation.

**TASK SIGNIFICANCE:**

The order-parameter relaxation time is an important parameter characterizing all dynamic processes in a phase transition. Accurate measurement of this quantity will allow a stringent test of the dynamic renormalization group theory which can be applied to predict the behavior of this quantity with no adjustable parameters.

**PROGRESS DURING FY 1994:**

Parts have been fabricated for the experimental probe. This probe is a duplication one that is currently in service for another project. A new post doctoral associate will join the group and begin conducting experiments in March 1995. Some calculations were performed to understand thermodynamics of helium under a sudden pressure quench.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 8/96

PROJECT IDENTIFICATION: 962-24-04-12

RESPONSIBLE CENTER: JPL

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Proceedings**

Chui, T.C.P. and Israelsson, U.E. "Heat capacity under pressure oscillations- a proposal to measure the order-parameter relaxation time in space." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

*Determination of the Correlation Length in Helium II in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. Russell J. Donnelly

University of Oregon

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this research is to measure finite size effects in the isobaric expansion coefficient near the lambda transition in liquid helium. We will measure the thermal expansion coefficient for liquid helium confined between parallel plates for a range of temperatures very near the lambda transition temperature (both above and below), a range of pressures from SVP to about 25 bar, and a range of plate separation distances.

**TASK DESCRIPTION:**

We will measure the dielectric constant of helium confined between parallel plates as a function of temperature at constant pressure. Using the Clausius-Mossotti relation, the density and thus the expansion coefficient of liquid helium will be calculated. The experimental method involves two measurements (at a given temperature) of the balancing ratio of an audio-frequency ratio-transformer capacitance bridge, one with the sample capacitor empty and then one with it filled with liquid helium. Appropriate division of these ratios then yields directly the dielectric constant at that temperature.

The capacitor used to measure the dielectric constant is a parallel-plate design operated as a three-terminal device in a 1-kHz ratio-transformer bridge. The spacing between the electrodes is determined by a precision shim which can be easily changed. An identical capacitor is also mounted on the experimental platform and is operated empty as a reference capacitor. We expect to vary the thickness of the shims between 5 microns and 50 microns.

Initially germanium thermometry will be used for temperature control and measurement. This will allow us to easily cover a wide range of temperature and to gain familiarity with the experiment. At this stage we will want to reconcile our results with older, published data. High-resolution measurements will be made after installing a paramagnetic salt thermometer identical to that used by John Lipa in his lambda-point heat capacity experiment (LPE), which successfully flew on STS-52 in October of 1992.

**TASK SIGNIFICANCE:**

Finite size effects are manifested as a rounding of the divergence in thermodynamic functions near a critical point as the correlation length increases toward the system size. We can thus test renormalization group theory predictions, universality assumptions, and boundary conditions.

**PROGRESS DURING FY 1994:**

The bulk measurements of density and expansion coefficient have been completed and will appear in J. Low Temp. Phys., vol 98, nos. 1/2, 1995. This work has identified interelectrode stray capacitance as the major source of error affecting the many experiments measuring dielectric constant of helium, and helps to explain the wide discrepancy in reported values of the deduced density over the last several decades. We are presently working with Dr. John Lipa of Stanford University to ascertain the possibility of taking the cryostat to Stanford to be fitted with high resolution thermometers there and then returned to Oregon for use in measuring density in finite-size gap capacitors in support of the CHEX experiment. Our experience with the bulk measurements should be invaluable in eliminating sources of error which would ruin any finite-size experiment.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Degrees: 0

MS Degrees: 0

PhD Degrees: 4

TASK INITIATION: 10/89 EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-24-04-04

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Niemela, J.J., and Donnelly, R.J. "Density and dielectric constant measurements in liquid helium." The Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

*Nucleation of Quantized Vortices from Rotating Superfluid Drops*

PRINCIPAL INVESTIGATOR: Prof. Russell J. Donnelly

University of Oregon

CO-INVESTIGATORS:

J. Niemela  
W-K Rhim

University of Oregon  
Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

The objective of this research is to study the nucleation of quantized vortices in helium II by investigating the behavior of rotating droplets of helium II in a reduced gravity environment.

**TASK DESCRIPTION:**

Two methods well-suited for levitating the helium drop in the near vacuum environment are electrostatic and/or magnetic levitation. A pure electrostatic scheme requires active feedback control, while a purely magnetic levitation requires large fields. A hybrid system is probably the best choice. Rotation can be accomplished by coupling to a charge distribution on the drop surface. We will initially use purely electrostatic levitation for studying drops. The required charging of the drops can be accomplished by forming the drops around a sharp electrode tip held at a high voltage. Film flow of helium II can be utilized to create drops at the bottom of a suitable container which can be filled by a fountain pump and situated above a pair of capacitor plates having an appropriate voltage difference between them.

**TASK SIGNIFICANCE:**

Nucleation phenomena, in general, are fundamental to many fields of physics and engineering. In the case of a rotating superfluid drop it will be possible to produce a state of zero nucleation, analogous to growing a perfect defect-free crystal. It should also be possible to add a controlled impurity to cause nucleation of a quantized vortex line in the drop. At low enough temperatures, this will be a pure quantum mechanical tunnelling phenomenon. At higher temperatures it should be possible to see thermally activated nucleation taking over, for a demonstration of nucleation under more familiar classical conditions. In conventional systems it is evident that vortex lines come from some preexisting source, probably vortices trapped by pinning sites on the walls. While this kind of source of vorticity is undoubtedly important, it is not as fundamental as the "extrinsic nucleation" problem where vortex line appears when none was present before.

**PROGRESS DURING FY 1994:**

We have improved the drop forming process to have variable control over the drop rate using a small uniformly distributed heater on the film flow vessel. The upper limit is determined by the smallest perimeter of the vessel above the free surface of helium contained in it, which for 1 mm size diameter drops produced from a nominally 1 cm diameter vessel is about 1 drop/s. The use of various tapers at the tip of the film flow vessel enables us to vary the drop size. We have produced both positive ions (ionized helium atoms) and negative ions (electron bubbles) using sharp tungsten field ionization or emission tips respectively. The best tips produce negative ions at a threshold voltage of about 400V and positive ions at a threshold of approximately 1500 V. Currents are in the range of picoamps for pure ionization or emission, but can increase to microamps by increasing the voltage enough that a vapor bubble forms around the tungsten tip. The tips are tested in the bath and the presence of ions is indicated either by observing the current flowing from a collector plate to an electrometer or by observing an "electric fountain" which forms in response to an ion stream. The amount of current measured for pure ionization is sufficient to charge the drops of 1 mm diameter, and at a rate of 1 drop/s. Very small micron size droplets have been briefly levitated, but in an uncontrolled manner, by picking up charge from a glow discharge of the tips or an arcing discharge through the helium vapor. We are currently working on charging single large diameter drops by pulsing the voltage to the ionization tips to avoid secondary cascading effects in the vapor which "burn out" the

tips. We have completed a theoretical study of the equilibrium configuration of a single vortex in a freely rotating superfluid drop (JLTP Vol. 98, Nos 1/2, 1995) and are working on extending these calculations to include multiple vortices.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-12

RESPONSIBLE CENTER: JPL

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BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Proceedings**

Bauer, G., Donnelly, R.J., and Vinen, W.F. "Vortex configurations in a freely rotating superfluid drop." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

Donnelly, R.J. "Superfluid Drops: An Overview." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

*Kinetic and Thermodynamic Studies of Melting-Freezing of Helium in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Charles Elbaum

Brown University

CO-INVESTIGATORS:

J.M. Kosterlitz

Brown University

**TASK OBJECTIVE:**

The objective of this project is to study, experimentally and theoretically, the effects of gravity on the melting-freezing transitions, including kinetic processes and the equilibrium shape of solids. The research is carried out on helium, whose unique properties render such investigations possible on a time scale consistent with experiments in space, under microgravity conditions. Indeed, morphological changes of the solid-liquid interface (i.e., the "surface" of helium) generally occur fast enough to satisfy the time constraints mentioned above.

**TASK DESCRIPTION:**

An optical system with special lighting applied to a growth cell contained in a liquid helium dewar allows viewing of the solid-liquid interface (SLI) and of crystal shapes. Rapid image capture equipment allows recording of the evolution of the SLI and of the crystal shapes in response to changes in temperature and pressure.

**TASK SIGNIFICANCE:**

These studies are addressing a number of fundamental questions, especially as they relate to the effects of gravity. These questions include the kinetics of first order phase transitions, the critical behavior in the evolution of crystal shapes as they approach equilibrium, faceting-roughening phenomena on various surfaces, relative and absolute values of interfacial free energy for different crystal faces, and the minimization of a system's free energy subject to various constraints. Furthermore, many applications should benefit from a deeper understanding of the above phenomena, among them crystal growth, surface configurations, sintering, and surface reactivity.

**PROGRESS DURING FY 1994:**

In our quarterly report submitted in May, 1994, we discussed our observations and some preliminary conclusions on crystal growth of body-centered cubic (b.c.c.) helium-four. These studies have continued, with the addition of nucleation, growth, and morphology of hexagonal close-packed (h.c.p.) crystals of helium-four.

We have extended these investigations to cover wider ranges of temperature, pressure, and rates of crystal growth. Our observations on both b.c.c. and h.c.p. helium four can be summarized as follows.

**I. b.c.c. solid helium-four**

In the temperature,  $T$ , and pressure,  $P$ , range of existence of the b.c.c. helium-four phase ( $1.43\text{K} < T < 1.76\text{K}$  and  $26.1\text{ atm} < P < 30.1\text{ atm}$ ), this solid behaves, in many respects, like viscous fluid. It should be emphasized that b.c.c. solid helium is characterized by translational symmetry and long range order typical of ordinary solids, as demonstrated by x-ray diffraction. Furthermore, the solid supports high frequency elastic shear waves and its smallest elastic shear modulus, measured at high frequency, is of the same order of magnitude as the longitudinal modulus. In particular, it is found that b.c.c. crystals formed at the top of a container filled with liquid (superfluid) helium, deforms under gravity into a pseudo-spherical shape. This observation indicates that the solid-liquid interfacial tension is essentially isotropic, in agreement with earlier findings. The time constant involved in this deformation (spherical shape acquisition) is of the order of a few seconds. The mass transport mechanism involved in this process is now being investigated. Our preliminary conjecture is that quantum mechanical tunneling of defects (primarily vacancies) is the predominant mechanism. Additional experiments to test this hypothesis are in progress.



II. The study of h.c.p. helium-four crystal growth from the normal fluid yields results in sharp contrast to those found for the b.c.c. case. Indeed, the shapes of h.c.p. crystals and the characteristics of the solid following solidification are remarkably different. In particular there is no evidence of the solid undergoing any measurable deformation under gravity, within the observation times available; this restricts any possible deformation rates to at least three orders of magnitude lower values than what is observed in the b.c.c. solid.

Another important factor emerging from the comparison of the behavior of b.c.c. and h.c.p. helium-four during and immediately following solidification concerns the effects of temperature on the processes under study. As mentioned above, the solidification of the h.c.p. phase took place from normal fluid, i.e. at higher temperatures than those of the b.c.c. phase. There is, however, no evidence of mass transport resulting in observable flow and deformation of the solid, while these characteristics are very prominent in (the lower temperature) b.c.c. phase. This indicates that the flow processes are not the same in the two phases and that if they are thermally activated, their activation energies are drastically different. Moreover, since the flow rates appear to be independent of temperature in the b.c.c. phase (over the limited range investigated), our tentative conclusion is that quantum mechanical tunneling of vacancies is the predominant mechanism of mass transport in the deformation of b.c.c. helium-four.

The theoretical studies carried out since the last report consist of two parts. The first consists of numerical simulations of growth of a solid from a liquid in the presence of impurities which tend to concentrate at the interface. This was modelled by modifying the free energy  $F(f, c)$  by adding a term that is a function of the conserved impurity concentration,  $c$ , and the order parameter  $f$  (-1 in the bulk liquid, +1 in the solid). The function was chosen to make  $c$  large at the interface when the gradient of  $f$  is large. Simulations were performed with initial conditions corresponding to a uniformly undercooled liquid. The results are in accord with our most naive expectations that this new interface term is just equivalent to reducing the stiffness constant or surface tension at the interface so no qualitative differences were observed. The interface became rougher and more convoluted but this is just a quantitative change and no new and unexpected effects were observed.

Some progress has been made on the problem of dealing with advection of mass from the liquid to the solid and the density difference between the two phases. Since experiments indicate that the b.c.c. phase of solid  $^4\text{He}$  allows for mass flow, the superfluid/b.c.c. solid system can be treated as a binary fluid mixture where the kinematic viscosity of the superfluid vanishes and is large in the solid. There is also a 10% density difference between the two phases, so gravity has significant effects. We have constructed a free energy functional  $F(r, f)$  and dynamical equations accounting for advection. We are intending to carry out numerical analysis of these equations starting from initial conditions for a uniformly undercooled superfluid and study the evolution of the solid. Gravitational effects can be included in our equations of motion. This constitutes a fairly realistic description of the solidification process.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-13

RESPONSIBLE CENTER: JPL

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BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Proceedings**

Elbaum, C., and Kosterlitz, J.M. "Kinetic and thermodynamic studies of melting-freezing of helium in microgravity." Proceedings of the NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

*Theoretical Influence of Microgravity on Critical Fluid Measurements*

PRINCIPAL INVESTIGATOR: Prof. Richard A. Ferrell

University of Maryland

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

This endeavor is tailored to provide theoretical support for these current critical fluid microgravity Space Shuttle experiments: the Critical Fluid Light Scattering Experiment (ZENO), the LeRC Critical Fluid Thermal Equilibration Experiment (CFTE), and several other microgravity experiments are now in the definition phase.

**TASK DESCRIPTION:**

The science activity will examines six areas; three each for classical fluids and three each for super-fluid Helium. Regarding classical fluids, they are:

1. Short and long time scale equilibration driving forces.
2. The anomalous dimension critical exponent of the density fluctuation correlation length.
3. Shear viscosity near the liquid-vapor critical point .

Regarding super-fluids, they are:

1. A better prediction of the thermal conductivity temperature scaling.
2. The frequency dependence of the shear viscosity.
3. Theoretical insight into the unexpected temperature dependence of the second-sound velocity of <sup>4</sup>He near its Lambda transition.

This effort will employ a graduate student, a part-time post-doctoral fellow, and a visiting faculty fellow to assist in this work.

**TASK SIGNIFICANCE:**

The results of the proposed study will both aid the interpretation of the data and demonstrate the need for the data from the objective microgravity experiments, confirming science conclusions. The conclusions will greatly improve the cost effectiveness of the science from identified flight experiments.

**PROGRESS DURING FY 1994:**

This task was extended only slightly into FY94 to allow administrative closeout. All objectives were accomplished by the end of FY93 and reported previously.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	2

TASK INITIATION: 1/91 EXPIRATION: 1/94

PROJECT IDENTIFICATION: 962-24-05-32

NASA CONTRACT No.:NAG3-1180

RESPONSIBLE CENTER: LeRC

*Condensate Fraction in Superfluid Helium Droplets*

PRINCIPAL INVESTIGATOR: Prof. J. Woods Halley

University of Minnesota

CO-INVESTIGATORS:

C. Giese

University of Minnesota

C. Campbell

University of Minnesota

K. Goetz

University of Minnesota

**TASK OBJECTIVE:**

The scientific goal of the proposed project is to obtain information about the condensate fraction in superfluid helium four by studying elastic scattering of helium atoms from a freely floating macroscopic sphere of the fluid.

**TASK DESCRIPTION:**

During the second year we have initiated experiments on the use of  $C_6$  covered surfaces to partially suspend He for a ground based experiment. We are making computational studies of various magnetic suspension methods. Many body calculations of transmission rates in the experiment have reached the stage of explicit numerical simulation, which is under way in collaboration with S. Chin of Texas A and M.

**TASK SIGNIFICANCE:**

The condensate fraction of the superfluid helium wavefunction is the microscopic manifestation of bose condensation which is universally believed to be the origin of the fluid's superfluid properties (as originally proposed by London more than 50 years ago). If successful, the experiment would be important because direct experimental study of the condensate fraction has proved extremely elusive. Only neutron scattering experiments give direct information and interpretation of these has proved difficult.

Our basic idea is that in a microgravity environment, it will be possible to do a tunneling experiment (analogous to a Josephson tunneling experiment in some respects) in order to study the condensate. We envision sending pulses of gaseous helium atoms at one side of a suspended sphere of superfluid helium four and detecting helium atoms emerging in coincidence from the other side of the sphere.

**PROGRESS DURING FY 1994:**

The objective of this project is the completion of a ground based study of the scientific and technical feasibility of an experiment in which the presence and nature of the long range quantum coherence (condensate) in superfluid helium four is detected. Pulses of gaseous helium will be fired at a suspended droplet of superfluid helium four and the resulting emission of helium atoms will be detected.

Theoretical results from the previous fiscal year are reported in Halley and Campbell (PRL 1993), Campbell (JLTP 1993), Halley (JLTP 1993), and Halley (Physica 1994). This work suggested the existence of a large effect but also clarified the need for a more complete many body analysis of the problem. Analytical aspects of this analysis have been completed in the past year and numerical work has been undertaken to make the needed variational calculations with post doctoral visitor Sang Hoon Kim.

The first ground based experiment, undertaken in collaboration with I. Silvera of Harvard University, was finished during this fiscal year. In this experiment, pulses of helium were fired at the horizontal surface of liquid helium at one end of a U-shaped copper tube about 3 in in length and 0.25 in in diameter. A detector at the horizontal liquid helium surface at the other end of the tube can detect re-emitted helium atoms. Mark Williams of this group spent several months in Cambridge working on this experiment. The experiment, which was not ideal in several respects

with regard to observing the effect, did not show evidence for prompt re-emission of helium atoms from the surface at the remote end of U tube.

We are engaged in studies for two further types of ground based experiments. M. Williams is carrying out an experiment at Minnesota to test the feasibility of a method for producing two parallel helium surfaces using the fact that cesium surfaces are not wet by superfluid helium four. A capacitive bridge for measuring liquid levels has been constructed and a preliminary experiment testing this idea will be run very soon.

Magnetic suspension has often been suggested as a method for droplet suspension of liquid helium in a microgravity environment. Experiments at Brown are currently under way to achieve magnetic suspension of helium droplets on earth using superconducting magnets. As an alternative J. Schmidt of this group has initiated a study of the possibility of using permanent magnets for this purpose. If it can be achieved this method would have great advantages with respect to simplicity and expense. It may even be possible to achieve suspension of helium droplets on earth using permanent magnets. To study these possibilities, Mr. Schmidt has been developing computer codes for determination of the optimum configuration of permanent magnets.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-14

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Campbell, C. E. Remnants of bose condensation and off-diagonal long range order in finite systems. J. Low Temp Phys., Vol 93, 907 (1993).

Halley, J. W. Structure of the condensate and some possible ways to observe it. J. Low Temp. Phys., Vol. 93, 893 (1993).

Halley, J. W. A novel approach to the observation of the condensate fraction in superfluid helium-four. Physica, Vol B97, 175 (1994).

#### Proceedings

J. W. Halley "Theoretical studies of manifestations of long range quantum coherence in <sup>4</sup>He in atom-droplet interactions." NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

J. W. Halley "Ground based experiments to observe long range quantum coherence in superfluid <sup>4</sup>He using atomic beams." NASA/JPL 1994 Microgravity Low Temperature Physics Workshop.

#### Presentations

Campbell, C.E. "Recent developments in liquid helium-four ." Eighth International Conference on Recent Progress in Many Body Theories, August 1994.

Giese, C. "A novel approach to the observation of the condensate fraction in superfluid helium-four." Eighth AIAA Symposium on Microgravity Science, Reno, Nevada, January 10-13, 1994.

Halley, J.W. "Formulations of the many body atom-droplet scattering problem." Workshop on Future Directions of Quantum Many Body Theory, January 14-16, 1994.

Halley, J.W. "Looking for new experimental manifestations of long range quantum coherence in superfluid Helium-4." Physics Colloquium, North Dakota State University, Fargo, ND, February 2, 1994.

Halley, J.W. "Looking for long range quantum coherence in superfluid helium." Physics Seminar at St. Olaf College, Northfield, MN, March 23, 1994.

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Halley, J.W. "Evaluating experimental ideas for observing long range quantum coherence in superfluid helium." Invited talk at the American Physical Society Washington meeting, April 19, 1994.

Halley, J.W. "Looking for long range quantum coherence in superfluid helium." Condensed Matter Theory Seminar, Ohio State University, Columbus, OH, April 18, 1994.

Halley, J.W. "Evaluating experimental ideas for observing long range quantum coherence in superfluid helium." Workshop on Quantum Fluid Clusters, Ringberg, Schloss, Kreuth-Oberhof, Bavaria, June 12-15, 1994.

Williams, M. "Ground based experiments on quantum coherence in helium-four." NASA Graduate Student Researchers Program 1994 Annual Symposium, Washington, DC, May 11-13, 1994.

*Ultra-Precise Measurements with Trapped Atoms in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Dr. Daniel J. Heinzen

University of Texas, Austin

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The task objective for FY94 is to trap laser-cooled Cs atoms in an "optical box" atom trap. During our preliminary work, we have already demonstrated the trapping of  $10^7$  Cs atoms at a temperature of 100  $\mu$ K in a magneto-optical atom trap (MOT). During the upcoming year we plan to design and to implement the lasers and optics required to generate an "optical box", which will be formed by six intersecting, elliptically focussed laser beams. We also plan to demonstrate that atoms from the MOT can be confined to the interior of this box. This constitutes the first step to realize our ultimate goal, which is to develop instruments and techniques that can be used to carry out precise measurements with atoms trapped in such a box, in a zero-gravity environment.

**TASK DESCRIPTION:**

In order to demonstrate the "box" trap, the Cs atoms will first be captured in a magneto-optical atom trap (MOT). This trap can easily capture about  $10^7$  atoms into a 1 mm<sup>3</sup> volume directly from a very dilute room temperature Cs vapor. The Cs atoms will then be released from the MOT, and begin to fall under the influence of gravity. At the instant they are released, the optical box trap will be turned on, thus capturing a large fraction of the atoms from the MOT. The box trap will be formed by six laser beams of elliptical cross section, that are tuned to the high frequency side of a Cs optical resonance line. These beams will be generated from a Ti:sapphire laser, prism beam expanders, and focussing optics. They will be intersected so as to form a cubical surface of light. Atoms striking the "walls" of the cubical surface will experience a repulsive optical dipole force that confines them to the interior of the box.

**TASK SIGNIFICANCE:**

Precision atomic resonance devices have tremendous practical and scientific importance. An important example is the atomic clock, which has widespread applications to navigation, geophysical measurement, and astrophysics. Precise tests of fundamental scientific principles such as time-reversal symmetry are also carried out with such devices. Substantial advances in the accuracy of these devices are therefore of great importance. One very promising avenue is to make use of ultracold, laser-cooled atoms. These atoms can move as much as 10,000 times more slowly than the atoms in conventional devices. This very low velocity can in principle substantially increase the accuracy of an atomic resonance device. This is because certain measurement errors are proportional to the velocity of the atoms. Unfortunately, gravity seriously limits the usefulness of laser-cooled atoms for this purpose, because it quickly accelerates the atoms to back to high velocities. Thus, only in the gravity-free environment of space can the full potential of these ultracold atoms be realized. Our goal is to develop techniques that could be used to produce and store a large number of ultracold atoms in the gravity free-environment of space. If successful, atomic resonance devices based on these techniques could lead to dramatic increases in accuracy.

**PROGRESS DURING FY 1994:**

The start date for this project was Sept. 7, 1994, and only a few weeks have elapsed since this date. However, some initial work on this project was carried out prior to the start date. During this time, we have built a magnetically shielded magneto-optical atom trap, and used it to trap  $10^7$  Cs atoms at a temperature of about 100  $\mu$ K. The trap consists of a Cs vapor cell that is evacuated to a pressure of  $10^{-8}$  torr with an ion pump. Laser beams from two frequency-stabilized, grating-tuned diode lasers are used in conjunction with a quadrupole magnetic field to trap and cool that atoms from the background Cs vapor. We are currently carrying out Zeeman resonance experiments on these atoms, and are designing the optics to produce the "box" trap discussed in the task objective.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-24-08-14

NASA CONTRACT No.: NAG8-1090

RESPONSIBLE CENTER: MSFC

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### *Dynamic Measurement Near the Lambda-Point in a Low-g Simulator on the Ground*

PRINCIPAL INVESTIGATOR: Dr. Ulf E. Israelsson

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

R.V. Duncan

Sandia National Laboratory

#### TASK OBJECTIVE:

The objective of this work is to perform dynamic measurements on a short cylindrical sample of helium very near the lambda-point in an effective gravity environment of about 0.01 g. Dynamic conditions will be created by passing a heat current through the sample. The effective low-gravity environment will be created by applying a magnetic field gradient which closely cancels hydrostatic pressure differences in the sample. Specifically, the reduced gravity conditions will enable a test of theoretical predictions of the effect of small heat currents on the nature of the lambda-transition and will allow probing of the interface region between co-existing normal and superfluid portions of the fluid. These measurements are not possible to perform in a regular lab environment on the earth due to the influence of hydrostatic pressure effects and the need to apply large heat currents to overcome hydrostatic effects, tending to perturb the fluid sufficiently to render the measurements questionable. The suppression of the lambda-transition due to heat currents will also be investigated at lower values of the heat current than possible in a one-g environment. The magnet will be procured from a magnet winding company and installed in a thermal platform under construction at JPL. Melting curve thermometers, which can operate well in strong magnetic fields, will be used for high resolution thermometry.

#### TASK DESCRIPTION:

The magnet will be designed and constructed by a magnet winding company. An experimental cell will be constructed with attachment points for melting curve thermometers to enable high resolution thermometry to be performed in the high field conditions of the experimental cell. The melting curve thermometers will be constructed at Sandia under a sub-contract to JPL. A high performance thermal platform will have the experimental cell and the magnet installed into it for performing the measurements. A vibration isolated and magnetically shielded helium dewar will be used to cool the thermal platform in order to minimize noise generation and improve the fidelity of the collected data.

#### TASK SIGNIFICANCE:

Recent investigations of the influence of an applied heat current on the properties of helium near the superfluid transition have revealed many new phenomena. Agreement with theories based on scaled mean field calculations and dynamic renormalization group calculations is not good. The disagreement may stem from the fact that theories assume zero-gravity conditions, while experiments are performed in a one-g environment. To overcome the influence of gravity on properties near the transition in a heat current, large values of heat current are required which has detrimental effects on the very properties in need of study. It has also been predicted that imposition of a heat current will change the very nature of the lambda-transition from continuous to first order. Investigating these phenomena in a simulated low-gravity environment would enable lower heat currents to be used and would enable observation of phenomena washed out by gravity effects.

#### PROGRESS DURING FY 1994:

The high field gradient superconducting magnet was delivered from the manufacturer in FY 94. Prior to delivery, the magnet was tested at the vendor facility to verify that all requirements had been met. The magnet was successfully operated up to 16.5 tesla at 2.2 K without quenching for a maximum field times field gradient product of 23 T<sup>2</sup>/cm. This is exceeding the requirement by nearly 10%. At JPL the magnet was installed in the thermal platform, cooled down, and performance tested. We experienced some quenching problems, releasing about 150,000 Joules of energy, when operating the magnet at near full field. The quenching problem was traced to a design flaw



with the "home made" current leads which we now believe to be solved. Field profile data was gathered using a hall probe device which was movable along the axis of the magnet. A second probe was used slightly off-axis to verify the performance over the entire experimental region.

The design and component fabrication of the thermal conductivity cell to be used in the experiments was also completed. The cell is 0.5 cm long and 0.5 cm in diameter and is made of stainless steel with copper end plates. The stainless steel side wall is 0.005 cm thick and has two circumferential temperature probes spaced evenly along the length of the cell. Final assembly of the cell is underway.

Six melting curve thermometers complete with reference capacitors have been constructed. Four of these thermometers will be used to measure the temperature of the two mid-plane thermometers, the top copper end plate, and the heat current control platform to near nanokelvin resolution.

To be able to better plan and analyze the low gravity simulator experiments, we have developed computer code to numerically simulate a one-dimensional thermal conductivity cell of helium near the lambda point. The basic time dependent heat flow equations and the known thermodynamic properties of helium at the lambda point were used. The effect of gravity was included by varying the spatial distribution of the local transition temperature in the cell accordingly. The results clearly demonstrate that a reduced gravity environment more closely achieves a quasi-static experimental condition.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-04-09

RESPONSIBLE CENTER: JPL

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#### Presentations

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Larson, M., and Israelsson, U.E. "The lambda point in a low-g simulator - progress report." The 1994 Microgravity Low Temperature Physics Workshop, Washington D.C., January, 1994.

### *Turbidity of a Binary Mixture Very Close to the Critical Point*

PRINCIPAL INVESTIGATOR: Prof. Donald T. Jacobs

The College of Wooster

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

This experimental activity is intended to measure the light transmitted through a near critical fluid consolute point of a density matched binary mixture.

#### TASK DESCRIPTION:

1. Develop a room temperature thermal control system for the sample that enables temperature control inside of  $\pm 3\mu\text{K}$ .
2. Assemble and use a phase sensitive detection system to detect the small light intensity variations expected close to the consolute point.
3. The turbidity data will be reduced according to the formalism of P. Calmettes et al. (Phys. Rev. Lett. 28, 478 (1972));.
4. Undergraduate students will assist Prof. Jacobs, making his activity a strong educational experience for the students.

#### TASK SIGNIFICANCE:

The resulting data will be used to confirm and quantify a small exponent " $\eta$ " in a denominator " $\{1+(q\xi)^2\}^{-\eta}$ " that describes the correlation length dependent distribution of scattered light from near critical fluids. Also, "two-scale universality" can be tested from the data set.

#### PROGRESS DURING FY 1994:

The optical system can now resolve the small light intensity variations anticipated in this experiment. The optical components rest on a vibration-isolated optics table. Using an intensity stabilized laser beam that is split and then chopped into a reference and signal beam, the stability of measured intensities is less than the requisite 0.1 percent. Lock-in amplifiers provide the sensitive detection of the photodiode signals.

The cell was redesigned to reliably seal the fluids yet allow light to pass through a 2mm thick slab of fluid. Once the critical composition for the liquid mixture is loaded into the cell, it will be suspended in a series of nested cylinders, each of which will stabilize the temperature fluctuations of the adjacent outer stage by a factor of 100 using an active temperature controller. The temperature controllers have been built and tested and the temperature sensors have been calibrated.

The computer software that accesses the instruments used in the experiment has been written in the language LabVIEW. Each portion of the program has been tested and debugged and the main controlling program that links the many sub-elements is being developed.

Associated experimental investigations that provide comparative information on other properties of binary liquids are being used to test "two factor universality." Coexistence curve and heat capacity data have been taken and are being analyzed for publication.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	7	BS Degrees:	2
MS Students:	0	MS Degrees:	0
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 1/93    EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-74

NASA CONTRACT NO. NAG 3-1404

RESPONSIBLE CENTER: LeRC

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### *Atom Interferometry in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Dr. Mark A. Kasevich

Stanford University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### **TASK OBJECTIVE:**

Our scientific objectives are: (1) to develop a rugged laser cooled source of atoms using DBR (distributed Bragg reflector) laser technology; (2) to use this source and these lasers to demonstrate coherent atom wavepacket manipulation techniques; and (3) to incorporate these techniques into an atom interferometer gravity gradiometer. We plan to experimentally study the performance of the prototype device to evaluate the feasibility of a space-based system.

#### **TASK DESCRIPTION:**

In year one of the grant period we will develop the laser sources and apparatus needed for the proposed experiments. We will employ standard laser cooling and trapping techniques with atomic cesium to create the cold atomic source. Our first experiments, to be carried out near the end of year one, will focus on demonstration and characterization of the proposed coherent atom manipulation techniques. In year two we will demonstrate and characterize an interferometer accelerometer - first in a low sensitivity regime in order to study potential systematic phase shifts and subsequently in a high sensitivity configuration to explore the potential accuracy and resolution of the device. Since vibrational noise will severely hamper the performance of an Earth bound accelerometer, we will switch to a more complicated gradiometer geometry which is far less sensitive to vibrational noise in the final stages of the proposed work.

#### **TASK SIGNIFICANCE:**

The convergence of recent advances in the field of laser manipulation of atoms with technological developments in the electronics/opto-electronics industry opens the possibility of a new class of experiments involving laser manipulated atoms in a microgravity environment. In the past five years, light-induced forces have been used to cool ensembles of atoms to temperatures below 1  $\mu$ K, providing researchers with a novel source of ultra-cold atoms. These laser cooled sources have revolutionized experimental atomic physics and have led to new classes of precision time standards and inertial sensors. Application of these techniques in a microgravity environment could result in robust gravity gradiometers and gyroscopes with sensitivities exceeding current state-of-the-art devices by several orders of magnitude. Such instruments would have important applications in a number of fields. For example, satellite gradiometry studies yield important geophysical data concerning Earth and ocean dynamics. A satellite borne accelerometer/gravimeter used in conjunction with the GPS system could be used to obtain highly accurate maps of the global Earth gravity field. In addition, techniques developed for satellite interferometer sensors might also have terrestrial applications in, for example, mineral/oil exploration and navigation. Finally, with minor modification, the techniques could be employed to develop atomic standards with unprecedented accuracy.

#### **PROGRESS DURING FY 1994:**

This grant has recently been initiated, and budgeting arrangements are still in process.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-24-08-15

NASA CONTRACT No.: NAG8-1088

RESPONSIBLE CENTER: MSFC

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*Effect of Confinement on Transport Properties by Making use of Helium Near the Lambda Point*

PRINCIPAL INVESTIGATOR: Prof. John A. Lipa

Stanford University

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

## TASK OBJECTIVE:

The objective of this project is to study the effect of confinement on the thermal conductivity of helium near the lambda point. The thermal conductivity is a transport property most readily accessible to precision measurement, allowing the effects of confinement to be quantified. The extent to which the results could be improved in a microgravity environment will also be studied.

## TASK DESCRIPTION:

Dohm and collaborators have predicted the first order departures of the thermal conductivity from the bulk behavior as one approaches the lambda point. As the transition is approached, the relevant length scale increases dramatically, allowing the effect to be measured in conventional apparatus. Higher order, nonlinear effects are also predicted, but quantitative information is not yet available. We plan to measure these effects for confinement in parallel plate geometry as a function of plate separation. Earlier measurements indicated that the first order effect was different to that predicted. This needs to be verified, and higher order contributions need to be explored.

## TASK SIGNIFICANCE:

The results will be used to test the emerging theory of transport properties in confined geometries, which has application to physical and chemical processes near surfaces or in small channels.

## PROGRESS DURING FY 1994:

Funding for this research became available at the beginning of FY95. Progress to date included investigations of previous thermal conductivity data to look for possible finite size effects. Experiments with helium confined within gaps as small as 70 microns have been examined.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	3	PhD Degrees:	1

TASK INITIATION: 9/94 EXPIRATION: 8/96

PROJECT IDENTIFICATION: 962-24-04-14

RESPONSIBLE CENTER: JPL

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*Theoretical Studies of the Lambda Transition of Liquid  $^4\text{He}$* 

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PRINCIPAL INVESTIGATOR: Prof. Efstratios Manousakis

Florida State University

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

We study the critical properties of liquid helium near the superfluid transition temperature  $T_\lambda$  using recently developed numerical simulation techniques and finite-size scaling. In particular, we are interested in the scaling behavior of the superfluid density and the specific heat. We shall study different finite geometries, namely pure two-dimensional, pure three-dimensional and the crossover from two-dimensional to three-dimensional superfluidity in order to verify the validity of scaling and to determine the universal functions associated with scaling.

**TASK DESCRIPTION:**

A recently developed updating technique called cluster Monte Carlo, which eliminates the long-standing problem of critical-slowing-down will allow us to approach close to the lambda point for large size lattices and, thus, extract the critical exponents and scaling properties of the physical quantities of interest. We shall study the temperature and the finite-size dependence of the superfluid density and the specific heat. From these studies we can determine the critical exponent using finite-size scaling techniques.

In addition we shall determine the superfluid/normal phase boundary  $T_\lambda(h)$  for films of thickness  $h$ . We shall calculate the superfluid density as a function of the film thickness and we shall examine the validity of the finite-size scaling theory. We shall also calculate the specific heat as a function of  $h$  and this will be used to understand the results of the CHEx experiment.

Finally, the role of vortices and the Kosterlitz-Thouless scenario will be also examined in the course of this work. We shall calculate the renormalization group beta function for two-dimensional superfluids and we shall compare it to that predicted by the Kosterlitz-Thouless theory. In addition, we shall study with our simulation technique the intimate connection between the superfluid transition and the unbinding of vortices.

**TASK SIGNIFICANCE:**

The results of these studies are relevant and will be compared to the experimental measurements obtained from the lambda-point experiment (LPE) and to the confined helium experiment (CHEx).

**PROGRESS DURING FY 1994:**

Using the  $x-y$  model and a non-local updating scheme called cluster Monte Carlo, we calculated the superfluid density  $\rho$  and the specific heat  $c$  of a superfluid in a film geometry, i.e. on a finite lattice of size  $L \times L \times H$  (where  $L \gg H$ ). In this geometry the superfluid density shows a three to two-dimensional crossover behavior. This means that below a certain crossover temperature the helium film behaves as if it was infinitely thick and, thus, it exhibits three-dimensional behavior. Above the crossover temperature and still below the bulk critical temperature  $T_1$  the helium film shows two-dimensional behavior. Because of that the critical temperature is reduced, the superfluid phase transition occurs at temperatures  $T_c(H)$  smaller than  $T_1$ . These reduced temperatures depend on the thickness of the helium film. In order to determine the critical temperatures  $T_c(H)$  we applied the Kosterlitz-Thouless-Nelson (KTN) theory, which was formulated for purely two-dimensional helium films, to the quantity  $T/(H\rho)$ . Namely, by solving the KTN renormalization group equations for this quantity we were able to obtain the values  $T/(H\rho)$  in the limit as  $L$  tends towards infinity; thus, the dependence on the planar dimensions  $L$  of the lattice was completely eliminated and  $T/(H\rho)(H)$  is a function of the film thickness only. We found that for a fixed film thickness  $H$  the ratio  $T/(H\rho)(H)$  close to  $T_c(H)$  obeys a simple functional form. Fitting this functional form to the computed values

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for  $T_c(H)$  we found estimates for the critical temperatures  $T_c(H)$ . The detailed results of this work have been submitted for publication in Phys. Rev. B.

The crossover behavior for the specific heat  $c$  is much less pronounced. Lattices with  $L=60$  are large enough to represent the infinite film thickness limit. For the films with  $H=6,8,10$  we obtain a universal curve that relates the heat capacity for films of finite thickness to the heat capacity of infinitely thick films. We are in the process of comparing our universal function with experimental results. The results of this investigation will be submitted to Phys. Rev. B.

The computations described above were carried out on the x-y model with periodic boundary conditions in all directions. This ensures the homogeneity of the physical quantities along the film thickness. For such a system we verify the validity of the finite-size scaling theory as reported above. However, in experiments on thin helium films which test the finite-size scaling theory,  $^4\text{He}$  is supported by substrates which impose different boundary conditions on the film. It appears therefore necessary to investigate the influence of other boundary conditions on the scaling behavior of the superfluid density and the specific heat with respect to the film thickness. From these investigations we hope to gain insight as to why recent experiments on thin helium films seem to yield results contradicting the finite-size scaling theory.

We have repeated the calculations described above for the x-y model in a film geometry with staggered (Dirichlet-like) boundary conditions in the top and bottom layer of the film. The superfluid density develops a profile with the maximum value in the middle of the film. At the boundaries the superfluid density vanishes. We have identified the temperature region where the model displays two-dimensional behavior and have used the KTN theory to determine the critical temperatures  $T_c(H)$ . Furthermore our data for the superfluid density and the specific heat do not collapse onto one universal curve until we applied an effective critical exponent  $n=0.923$ . It seems that these preliminary results show the same situation here as was described by Rhee, Gasparini and Bishop who had to use an effective critical exponent  $n=1.14$  to achieve scaling of their data for the superfluid density with film thickness. However, we are in the process of trying to understand these results further.

The calculations reported above were performed on a heterogeneous environment of workstations which include DEC Alpha, Sun, and IBM RS/6000 workstations and on the Cray-YMP supercomputer and took several months of CPU time.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 01/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-15

RESPONSIBLE CENTER: JPL

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**Proceedings**

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Schultka, N., and Manousakis, E. "Finite size scaling in two-dimensional superfluids." APS Meeting, Pittsburgh PA, March 1994.

*Dynamics and Morphology of Superfluid Helium Drops in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. Humphrey J. Maris

Brown University

CO-INVESTIGATORS:

G. Seidel

Brown University

**TASK OBJECTIVE:**

The long range goal of our research is the study of the hydrodynamics of drops of superfluid liquid helium by means of microgravity experiments conducted in space. At the present time we are developing a series of earth-based experiments to levitate superfluid drops so that we can acquire data and experience that will be needed for the design of experiments in space.

**TASK DESCRIPTION:**

We are conducting a series of earth-based experiments to study the behavior of superfluid drops. We will develop a means to levitate helium drops in earth gravity, primarily by magnetic levitation. We will then investigate 1) how to inject and position drops in a microgravity chamber, 2) how to manipulate drops and to give them angular momentum, 3) how to observe accurately the vibrations and rotations of the drops, and 4) what drop sizes are best suited for the study of a variety of phenomena.

**TASK SIGNIFICANCE:**

The goal is to achieve data and experience critical for the design of experiments in space.

**PROGRESS DURING FY 1994:**

**Optical Levitation Experiment:** We have completed our experiments on optical levitation of superfluid helium-4. Drops of diameter approximately 20 microns were levitated using an optical trap formed by a focussed Nd:YAG laser beam of wavelength 1.06 micron. It was possible to levitate drops for as long as three minutes. We have carried out a detailed analysis of a number of phenomena observed during this experiment. Several factors that might control the rate at which the drops evaporated were analyzed, including laser heating, evaporation due to the curved surface of the drop, and non-equilibrium conditions inside the experimental cell. We performed detailed computer calculations of the potential energy in the vicinity of the optical trap. The experiment revealed that it was possible for several drops to be held in the trap at the same time without coalescence occurring. We considered a number of possible explanations for this remarkable result. A paper on the optical levitation experiment will appear in the *Journal of Low Temperature Physics*. In addition, the staff of *Scientific American* have written a short article about the experiment to be included in a forthcoming issue of their magazine.

**Magnetic Levitation Apparatus:** We have continued construction of the apparatus to magnetically-levitate helium. The magnet constructed by Oxford Instruments has been delivered. The large Janis dewar for the magnet has been mounted and the cryostat is under construction. The cryostat is mounted adjacent to a large optical table on which we can mount lasers for illumination and an optical recording system. This cryostat has been designed to be suitable for a variety of experiments on helium drops, at temperature down to 0.5 K.

**Fluid Mechanics of Drops in Micro-Gravity:** We have started a calculation designed to determine the morphology of a superfluid drop with angular momentum under micro-gravity conditions. The goal is to determine how the shape of the drop changes as the angular momentum is increased. For the moment we are restricting attention to consideration of the shape with lowest energy for a given angular momentum.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-16

RESPONSIBLE CENTER: JPL

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*Critical Transport Phenomena in Fluid Helium Under Low Gravity*

PRINCIPAL INVESTIGATOR: Prof. Horst Meyer

Duke University

CO-INVESTIGATORS:

F. Zhong

Duke University

**TASK OBJECTIVE:**

Ground-based experiments will be carried out to study the density equilibration process at constant average density  $\rho$  in a pure fluid, ( $^3\text{He}$ ), near its liquid-vapor critical point ( $T_c, \rho_c$ ) after a step change in temperature  $\Delta T$  of the container walls. Measurements are to be carried out for both the region above  $T_c$  (one phase) and below  $T_c$  (coexisting phases). Numerical simulations are to be performed of the density, temperature and pressure equilibration processes in  $^3\text{He}$  for the regime above  $T_c$ , both ground-based and under reduced gravity. The measurements are to be extended to binary ( $^3\text{He}$ - $^4\text{He}$ ) mixtures near their liquid vapor critical point ("plait point").

**TASK DESCRIPTION:**

Two flat, horizontal cells of somewhat different geometry are used in different series of experiments. The fluid is contained between two parallel flat OFHC copper plates with high thermal conductivity, kept at the same temperature that is regulated to within a few mK before and after the step  $\Delta T$ . In both cells, the fluid layer height is approximately 4 mm, the diameter is 30mm and the density is measured by two thin horizontal superposed capacitive sensors, spaced by 2 mm that record the dielectric constant. The density is then derived via the Clausius-Mossotti relation, and is recorded by both sensors,  $\rho_{\text{top}}(t)$  and  $\rho_{\text{bottom}}(t)$ , as a function time  $t$  after a programmed small temperature step of the cell walls. At temperatures above  $T_c$ , immediately after the step  $\Delta T$ , the fast density change from adiabatic energy transfer ("piston effect") followed by the slow stratification change at each sensor are recorded by computer. Experiments are carried out along several near-critical isochores and along several isotherms. Below  $T_c$ , the two sensors detect respectively the coexisting liquid and vapor phases. An induced temperature change in the cell walls will permit following the density evolution in both phases with time.

With help of the known scaled expressions for the static and transport properties of  $^3\text{He}$  above  $T_c$ , numerical simulations in one dimension are carried out to predict the temporal and spatial evolution of the thermodynamic parameters (density, temperature, pressure and their derivatives) and predict the asymptotic relaxation times. This simulation is to be done at arbitrary values of the gravitational acceleration  $g$ .

After completion of the  $^3\text{He}$  program, measurements and numerical simulations are to be extended to binary ( $^3\text{He}$ - $^4\text{He}$ ) mixtures.

**TASK SIGNIFICANCE:**

Such studies - both for pure fluids and binary mixtures - are very relevant to experiments on fluids under  $mg$  conditions, where investigations of static and dynamic properties near critical points are to be carried out. It is important to know how long a fluid system takes to approach closely enough thermodynamic equilibrium, and what are the basic mechanisms that control the equilibrium process. The numerical computations above  $T_c$  are to be compared with experiments. Simulations under microgravity conditions will be able to assess the permissible temperature ramping rate in experimental data taking that will enable measurements of critical properties in a quasi-equilibrium state. In the two-phase regime below  $T_c$ , little is known about the equilibration dynamics and the proposed experiments are expected to substantially help in understanding these processes.

**PROGRESS DURING FY 1994:**

During spring '92, the existing cryostat for work over a temperature range 1-5 K (previously used in the NASA-sponsored viscosity measurements) was modified to incorporate a temperature-regulated platform supporting the density equilibration cell. Electronic circuitry was installed for high-resolution stable temperature and dielectric constant measurement arrangement. Computer programs for temperature step control and automatic data acquisition and reduction were developed. The apparatus was tested over a period of several months, and data-taking routine was developed and gradually perfected. Initial density stratification data were taken in the one-phase regime along the critical isochore and also in the two-phase regime. Temperature steps of different sizes and sign were used and the stratification data were analysed in a preliminary fashion. Stratification time was found to diverge as the critical point is approached from both the single and the two-phase regimes. At a given temperature, it was found to be the same coming from either the colder or the warmer side. We expect to continue with data taking along other isochores and along isotherms, and hope to present a scaling scheme of our data.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 2/92 EXPIRATION: 1/94

PROJECT IDENTIFICATION: 962-24-02-01

RESPONSIBLE CENTER: JPL

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*Equilibration in Density and Temperature Near the Liquid-Vapor Critical Point*

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PRINCIPAL INVESTIGATOR: Prof. Horst Meyer

Duke University

CO-INVESTIGATORS:

F. Zhong

Duke University

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**TASK OBJECTIVE:**

Ground-based experiments will be carried out to study the density equilibration process at constant average density  $\bar{\rho}$  in a pure fluid, ( $^3\text{He}$ ), near its liquid-vapor critical point ( $T_c, \rho_c$ ) after a step change in temperature  $\Delta T$  of the container walls. Measurements are to be carried out for both the region above  $T_c$  (one phase) and below  $T_c$  (coexisting phases). Numerical simulations are to be performed of the density, temperature and pressure equilibration processes  $^3\text{He}$  for the regime above  $T_c$ , both ground-based and under reduced gravity. The measurements are to be extended to binary ( $^3\text{He}$ - $^4\text{He}$ ) mixtures near their liquid vapor critical point ("plait point").

**TASK DESCRIPTION:**

Two flat, horizontal cells of somewhat different geometry are used in different series of experiments. The fluid is contained between two parallel flat OFHC copper plates with high thermal conductivity, kept at the same temperature that is regulated to within a few mK before and after the step  $\Delta T$ . In both cells, the fluid layer height is approximately 4mm, the diameter is 30mm and the density is measured by two thin horizontal superposed capacitive sensors, spaced by 2 mm that record the dielectric constant. The density is then derived via the Clausius-Mossotti relation, and is recorded by both sensors,  $\rho_{\text{top}}$  and  $\rho_{\text{bottom}}$  ( $t$ ), as a function time  $t$  after a programmed small temperature step of the cell walls. At temperature above  $T_c$ , immediately after the step  $\Delta T$ , the fast density change from adiabatic energy transfer ("piston effect") followed by the slow stratification change at each sensor are recorded by computer. Experiments are carried out along several near-critical isochores and along several isotherms. Below  $T_c$ , the two sensors detect respectively the coexisting liquid and vapor phases. An induced temperature change in the cell walls will permit following the density evolution in both phases with time.

With help of the known scaled expressions for the static and transport properties of  $^3\text{He}$  above  $T_c$ , numerical simulations in one dimension are carried out to predict the temporal and spatial evolution of the thermodynamic parameters (density, temperature, pressure and their derivatives) and predict the asymptotic relaxation times. This simulation is to be done at arbitrary values of the gravitational acceleration  $g$ .

After completion of the  $^3\text{He}$  program, measurements and numerical simulations are to be extended to binary ( $^3\text{He}$ - $^4\text{He}$ ) mixtures.

**TASK SIGNIFICANCE:**

Such studies - both for pure fluids and binary mixtures - are very relevant to experiments on fluids under  $mg$  conditions, where investigations of static and dynamic properties near critical points are to be carried out. It is important to know how long a fluid system takes to approach closely enough thermodynamic equilibrium, and what are the basic mechanisms that control the equilibrium process. The numerical computations above  $T_c$  are to be compared with experiments. Simulations under microgravity conditions will be able to assess the permissible temperature ramping rate in experimental data taking that will enable measurements of critical properties in a quasi-equilibrium state. In the two-phase regime below  $T_c$ , little is known about the equilibration dynamics and the proposed experiments are expected to substantially help in understanding these processes.

## PROGRESS DURING FY 1994:

There have been three main projects in this period:

- A) Processing and analyzing temporal density evolution  $r$  data in  $^3\text{He}$  after a programmed temperature step of the cell. These data had been obtained throughout 1993.
- B) Developing and improving the computer simulation program that calculates the spatial and temporal evolution of thermodynamic coefficients of  $^3\text{He}$ .
- C) Writing a detailed paper describing both the experimental and the computed results for  $^3\text{He}$  above the critical point, and also writing papers for Proceedings.

A) Data analysis. In the experiments that had lasted approximately 12 months, very systematic measurements of the density equilibration were carried out both above and below the liquid vapor critical point ( $T_c$ ,  $r_c$ ,  $P_c$ ). During this lengthy data taking, measurements were performed of the density evolution in time  $r(t)$  at two superposed locations in a cylindrical cell after a programmed temperature step on the fluid container. This density evolution towards equilibrium was automatically stored in a computer. Experiments were carried out along several isochores, one of them being the critical one. Also experiments were performed along several isotherms both above and below the critical point below which both the liquid and the coexisting vapor phases could be sampled. Of this very large amount of data, those along the critical isochore and several isotherms have been processed and displayed graphically versus time. The superposed location of the two density sensors in the cell permitted the observation of the density stratification in the cell, under the effect of the earth's gravity.

B) Computer simulations. Together with the data analysis, the computer simulation of the temporal and spatial evolution of temperature  $T(z,t)$ , density  $r(z,t)$  and pressure  $P(z,t)$  has been significantly improved. This simulation, which is restricted to the region above the critical point (single phase), uses the published static and dynamic critical properties of  $^3\text{He}$  obtained in this laboratory, and calculates the equilibration of  $T$ ,  $r$  and  $P$  following the programmed temperature step of the fluid container. This is done for an arbitrary gravitational acceleration  $g$ . Hence the computer simulation can yield results for the density evolution that can be directly compared with experiments in a ground-based laboratory, and it also predicts the evolution of the coefficients during an experiments under microgravity conditions. Much effort was spent comparing experimental data and computations and the results can be briefly summarized as follows: 1) There is an impressive agreement between the observed and predicted amplitudes and shapes of the density evolution. In particular at temperatures well above the critical point, the predicted sharp spike resulting from the adiabatic energy transfer ("piston effect") was clearly observed and the predicted and observed amplitudes were in good agreement. The same good agreement was found for the amplitude of the stratification. 2) However there is a systematic discrepancy in the time scales involved. While the observed and predicted relaxation times agree far above  $T_c$ , there is a systematic departure as  $T_c$  is approached, whereby the computed relaxation time diverges more strongly than the experimental one. Close enough to  $T_c$ , the stratification causes a leveling off of the relaxation time curve versus  $(T-T_c)$ , both predicted and observed. Under microgravity conditions, the computer simulation shows that the relaxation time continues diverging. 3) Furthermore simulations in calculating the properties inside the fluid in the course of temperature ramping - both upwards and downwards were conducted, which gave remarkable results. In particular the rms density variation throughout the fluid was predicted as a function of ramping rate and temperature.

C) Manuscripts. Several drafts of a long and detailed manuscript "Density equilibration near the Liquid-Vapor critical point of a pure fluid: I Single phase  $T > T_c$ " have been produced, and we hope that the final paper will be ready for submission to Physical Review sometime by middle of November. A first draft of the sequel of this paper II- coexisting phases  $T < T_c$  has been written.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/94 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-20

RESPONSIBLE CENTER: JPL

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*Nonlinear Dynamics and Nucleation Kinetics in Near-Critical Liquids*

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PRINCIPAL INVESTIGATOR: Prof. Alexander Z. Patashinski

Northwestern University

## CO-INVESTIGATORS:

M. Ratner

Northwestern University

V. Pines

Case Western Reserve University

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TASK OBJECTIVE:

This grant activity is an early definition study to establish the theoretical foundations of the nonlinear response to strong perturbations in near critical fluids. This is a subset of a larger goal to develop an understanding of the many nonequilibrium states of strongly interacting systems. Critical fluids are to provide the experimental and theoretical model systems. This early definition work will be the basis for one or more future low gravity experiments.

## TASK DESCRIPTION:

The Principal Investigator will work on a description of the non linear formation and relaxation of inhomogeneities in liquid-vapor and binary mixtures near their critical points. The perturbations from homogeneity may occur from stable and unstable initial thermodynamic states. There is a need for the inhomogeneities to be large and long lived. Renormalization group formalism will be used to describe the non-Gibbsian distribution functions of the critical fluctuations. Three experiments have been suggested in the proposal. They are as follows:

1. Relaxation of large inhomogeneities to a one-phase final state.
2. Nucleation kinetics during the formation of a final two-phase state starting from an initial unstable state.
3. Nucleation kinetics and the influence of spinodal decomposition dynamics on domain nuclei in a state near the spinodal line.

## TASK SIGNIFICANCE:

This is fundamental research into the non linear understanding of nucleation phenomena. Its long term technological value rests on application to nucleation phenomena in a myriad of common phenomena at small space and time scales.

Broader fundamental knowledge of strongly interacting systems in non-equilibrium can open up new technologies. Our lack of knowledge in this area limits our current technological imagination.

## PROGRESS DURING FY 1994:

Since funding was initiated in June 1994, the investigators have quantitatively analyzed weak perturbations in the linear regime to confirm their formalism yields and adiabatic fast heat transfer. They are now advancing their efforts to apply the formalism to the non linear heat transfer in the vicinity of a strongly perturbing hot body in a very compressible critical fluid.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-07

RESPONSIBLE CENTER: LeRC

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*Precise Measurements of the Density and Thermal Expansion of  $^4\text{He}$  Near the Lambda Transition*

PRINCIPAL INVESTIGATOR: Dr. Donald M. Strayer

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

Dr. Talso Chui

Jet Propulsion Laboratory (JPL)

Prof. Nai-Chang Yeh

California Institute of Technology

Dr. Mark Lysek

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

The objective of this ground-based Annual NRA project is to demonstrate the value of high-precision density measurements in the study of the lambda transition of liquid helium. During the two years of Annual NRA funding, we shall demonstrate the capability to perform high-precision density measurements using superconducting cavities, applying high resolution thermometers (HRTs) for temperature control. We shall also demonstrate the ability to deconvolve nonuniformities caused by gravity from the density data.

**TASK DESCRIPTION:**

We shall employ superconducting microwave cavities having Q-values near  $10^{10}$ , operated in modes that have standing wave patterns that are axially symmetric and whose z-dependences are well known. Upon filling the cavity with liquid helium, and adjusting the temperature near to  $T_\lambda$ , measurements of the cavity resonant frequency will reflect the helium density. Very near the transition a normal-superfluid interface will form in the cavity: Moving this interface across antinodes of the standing wave pattern by high resolution temperature control will allow the density to be probed in the interfacial region. Measurements at many temperatures will provide density data for deconvolving the temperature dependence of the density from the gravity-induced effects.

**TASK SIGNIFICANCE:**

We expect that our earth-bound measurements will demonstrate the value of precision density measurements to exploration of the lambda transition, and to study of cooperative transitions in general. The preliminary measurements to be conducted in this Annual NRA task will provide useful information about future exploration of related experiments to be conducted in the microgravity experiments. The results will lead to applications of the technique to problems that include studies at many pressures, studies of nonequilibrium effects, or studies in confined geometries.

**PROGRESS DURING FY 1994:**

This task has very recently been provided with funds. Since that time, the design of a probe on which to perform the demonstration measurements has progressed through development of a rudimentary thermal model, plus the determination of required frequency resolution in the microwave components. Obtaining those microwave components is now underway, with all major pieces in hand, and only some smaller pieces yet to be procured. As stated in the proposal, an existing superconducting cavity and a cryoprobe already in Prof. Yeh's laboratory will be modified for use in these measurements. The thermal model will be improved, and then will be used to design the modifications to yield the necessary thermal stability on the platform.

The postdoctoral fellow who will perform the measurements is identified, has participated in the design of the microwave measurement system, and will be on board by the start of 1995.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 8/96

PROJECT IDENTIFICATION: 962-24-04-12

RESPONSIBLE CENTER: JPL

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### *Evaluation of Ovarian Tumor Cell Growth and Gene Expression*

PRINCIPAL INVESTIGATOR: Prof. Jeanne L. Becker, Ph.D.

University of South Florida

CO-INVESTIGATORS:

G.F. Spaulding  
R.H. Widen

NASA Johnson Space Center (JSC)  
University of South Florida (Tampa Gen. Hospital)

#### TASK OBJECTIVE:

Examine growth and gene expression of LN1 ovarian tumor cells cultured in the Rotating-Wall Vessel (RWV).

#### TASK DESCRIPTION:

A model for the growth of human ovarian tumor cells has been developed. This model allows for three-dimensional growth of LN1 ovarian tumor cells, facilitating the generation of tissue-like aggregates of cells exhibiting complex architecture. The cell populations generated during RWV culture morphologically and histochemically resemble those characteristics exhibited by the original patient tumor specimen from which this cell line was developed.

#### TASK SIGNIFICANCE:

*In vitro* growth of human ovarian tumor cells is not readily accomplished using traditional culture methodologies. Using the low shear, low turbulence conditions provided by the microgravity environment of the NASA RWV, we have developed a model which supports the culture of these cells under conditions that simulate *in vivo*-like growth. We have used this model to study growth characteristics in conjunction with changing patterns of gene expression and oncoprotein production occurring during complex cellular aggregate formation *in vitro*.

#### PROGRESS DURING FY 1994:

During this year, my laboratory has received two rotating-wall vessels, a slow-turning lateral vessel (STLV) and a high aspect rotating-wall vessel (HARV). We have become familiar with the care, set-up and operation of these vessels in our hands and the growth of LN1 in these vessels. During culture in the RWV, LN1 cells become highly metabolic, exhibiting greatly increased rates of glucose utilization during continued three-dimensional growth. Because of the greater oxygenation capacity of the HARV, the growth of LN1 cells in the HARV is superior to growth in the STLV. In conjunction with an increased rate of glucose utilization, LN1 also exhibits an increase in the percentages of cells undergoing DNA synthesis (S phase of cell cycle) and mitosis (G<sub>2</sub>+M phases) with time in HARV culture.

We have demonstrated that the LN1 cell line has the capacity to differentiate into multiple cell populations during three-dimensional culture in the RWV. These populations have been characterized both immunocytochemically and flow cytometrically. Recent studies have evaluated staining patterns of intermediate filaments and oncoproteins in LN1 cells grown in the RWV, as compared to cells present in the original tumor specimen from which LN1 was derived. Staining patterns for cytokeratin, vimentin and chondroitin sulfate present in LN1 grown three-dimensionally in the HARV were remarkably similar to that observed in the original tumor, in contrast to patterns observed in control Petri dish cultures and monolayer culture. LN1 cells grown in the HARV also demonstrated increased staining for TAG-72 carcinoma antigen and c-erbB2 oncoprotein, as exhibited by the original specimen.

LN1 cells in monolayer culture exhibit constitutive expression of mRNA and protein for c-erbB2, c-ras and mutant p53. Analysis of LN1 grown three-dimensionally also revealed constitutive expression of mRNA for c-erbB2, c-ras and mutant p53, although differential expression of the corresponding oncoproteins, p185<sup>c-erbB2</sup>, p21<sup>c-H-ras</sup>, p21<sup>c-K-ras</sup> and mutant p53 protein, was observed within individual cell populations generated during RWV culture. Additional studies have revealed that LN1 expression of TGF-beta is altered in RWV culture, relative to two-dimensional growth in monolayer culture.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 11/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-23-01-15

NASA CONTRACT No.: NAG-648

RESPONSIBLE CENTER: JSC

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*Crystallographic Studies of Proteins Part II*

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PRINCIPAL INVESTIGATOR: Dr. Daniel C. Carter

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

J. Ho (X. He)

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

This research involves the atomic structure determination of several protein structures. Key areas of study involve serum albumin structure and chemistry and HIV antibody complexes and structure. Aspects of this research generate flight experiment problems and contribute to facilities for the evaluation of flight experiment activities.

**TASK DESCRIPTION:**

The overall goal of this research is to utilize ground-based and microgravity-grown protein crystals to improve our understanding in two important areas of structural molecular biology. The first involves the determination of the definitive structure of serum albumin together with the chemical basis for the molecules' tremendous ability to bind and transport an immense variety of ligands throughout the circulatory system. The second area which is also broad in scope involves the structure determination of a series of human monoclonal antibodies expressed against the AIDS virus together with their respective antigen complexes. Both of these project areas are in an advanced stage where improvements in crystal quality will have significant impact on our understanding of the underlying chemistry.

Screens for optimum crystallization conditions or to determine crystallization conditions for new proteins will be conducted by the hanging-drop vapor-diffusion method. A Micromedics robotic crystal growth system is available to aid systematic surveys of pH, precipitant type, precipitant concentration, and protein concentration. The monoclonal antibodies expressed against the AIDS virus will be supplied by collaborator Professor Florian Rucker of The Institute of Applied Microbiology in Vienna, Austria. Cleavage of the antibody with papain or pepsin to produce the Fab fragments and subsequent purification will continue to be conducted. Antigenic peptides will be provided by Dr. Rucker and/or as a gift from IAF Biochemicals of Canada. X-ray diffraction data will be collected from both ground-based and flight crystals using a multi-wire area detector (Nicolet) and an imaging plate system (R-Axis) mounted on a Rigaku RU200 rotating anode generator. In favorable cases where the logistics can be arranged, diffraction data will be collected at synchrotron sources.

**TASK SIGNIFICANCE:**

High quality, single crystals are of tremendous value for a variety of industrial and research applications. Crystals of sufficient size and quality also provide invaluable avenues to understanding the detailed atomic structure and function of biological macromolecules and other substances. Efforts to produce higher quality protein crystals for application in x-ray crystallography have spawned numerous experimental approaches which range from the application of automated screening to the growth of protein crystals in microgravity.

**PROGRESS DURING FY 1994:**

The structure of *Schistosoma japonica* glutathione-S-transferase fusion protein was determined. This structure has several important implications:

1. The protein contains the principal portion of an epitope of gp41 of the human immunodeficiency virus type 1 which is recognized by a neutralizing antibody, 2F5.
2. The protein is from a parasitic flatworm which is the causative agent of schistosomiasis, the second leading cause of death and suffering in the world (malaria is primary). According to the scientific literature, this protein was a leading candidate for the development of a vaccine against this disease.

3. The protein is part of an important commercial protein expression system. We have proposed that fusing proteins which have proven difficult to crystallize to GST may provide a new and expeditious route to their structure determination. Verification is in progress.

The refinement of several structures is nearing completion. Cytochrome c5 from *Azotobacter vinlandii* has been refined to 1.9 angstroms resolution. HEW lysozyme grown in microgravity is refined to previously unreported high resolution. Additional high quality flight crystals are required to finish this work. A new myoglobin structure is solved and in the final stages of refinement. The nature of long-chain fatty acid binding to albumin was determined.

Several new and important proteins have been crystallized. Several manuscripts are in preparation.

Also, this year an authoritative review on the structure of albumin was published by Academic Press.

We completed the first year of two government/industry cooperative agreements. Both are interested in extending and/or expanding the agreements. Three additional agreements are under consideration.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-08-17

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

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*Microgravity Studies of Cell-Polymer Cartilage Implants*

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PRINCIPAL INVESTIGATOR: Dr. Lisa E. Freed

Massachusetts Institute of Technology (MIT)

## CO-INVESTIGATORS:

R. Langer

Massachusetts Institute of Technology (MIT)

G.F. Spaulding

NASA Johnson Space Center (JSC)

T.J. Goodwin

NASA Johnson Space Center (JSC)

D. Ingber

Harvard University

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**TASK OBJECTIVE:**

The long-term objectives of our current studies are: (1) to assess the effects of *in vitro* culture conditions, microgravity in particular, on tissue morphogenesis, (2) to correlate the characteristics of engineered tissues with bioreactor fluid dynamics in the form of physically and biologically sound mathematical models, and (3) to elucidate the mechanisms underlying the effects of microgravity on the structure and function of the engineered tissues. Related practical objectives are to optimize bioreactor design and develop operating strategies for cultivating tissues under conditions of simulated and actual microgravity.

**TASK DESCRIPTION:**

A simulated microgravity environment can be used to engineer cell-polymer tissue constructs, the size and shape of which are determined by an FDA approved, biocompatible, biodegradable scaffold with a defined three dimensional (3D) shape and structure. In particular, fibrous polyglycolic acid (PGA) can be seeded with isolated cells (chondrocytes) and cultivated in rotating bioreactors to make cartilaginous tissue constructs for *in vitro* studies of tissue morphogenesis and/or *in vivo* implantation.

**TASK SIGNIFICANCE:**

Engineering cartilage (i.e., tissue constructs grown *in vitro* using isolated cells and biomaterial scaffolds) can be used *in vivo*, to create subcutaneous neocartilage (in nude mice) and for joint resurfacing (in rabbits), and thus represents a biologically based therapy for repairing cartilage damaged by congenital defects, arthritis, or trauma. We have shown that the structure of engineered cartilage depends on hydrodynamic forces during *in vitro* cultivation; this is similar to the known effects of environmental forces on the tissue morphogenesis *in vivo*. For example, structural organization of bone and cartilage depends on the mechanical distribution of compressive and tensile stresses. Ground-based research utilizing rotating vessels (simulated microgravity) is expected to enhance our understanding the principles governing tissue morphogenesis. Space studies (actual microgravity) can further extend the operating limits of these vessels. The same approaches and methodologies can also be applied to other cell-polymer model systems, in order to engineer other clinically useful tissues.

**PROGRESS DURING FY 1994:****Specific Aim (1):**

Establish methods to culture chondrocytes in 3D synthetic, biodegradable polymer scaffolds in a rotating vessel to regenerate cartilaginous tissue.

Chondrocytes were uniformly seeded and cultivated on polymer scaffolds in two rotating vessels, the Slow Turning Lateral Vessel and the High Aspect Ratio Vessel (STLV, HARV). One week cell-polymer constructs grown in rotating vessels contained more GAG (total and per gram) and did not have fibrous outer capsules, as compared to control constructs grown in a previously established spinner flask system. These findings can be attributed to effects of bioreactor fluid dynamics, and indicate that differentiated cell function is promoted under conditions of simulated microgravity. As compared to natural bovine articular cartilage, 1 week cell-polymer constructs had three times more cells, 75% as much GAG, and 10% as much collagen per gram dry weight.



## Specific Aim (2):

Amplify chondrocytes isolated from small cartilage biopsy specimens to obtain the cell mass required to seed clinically sized polymer scaffolds.

Cells can be amplified using petri dishes (serially passaged monolayers) or bioreactors (cell spheroids), but these processes can be associated with loss of differentiated cell function (e.g., secretion of type I instead of type II collagen). We are developing an ELISA to quantitate the type the collagen extracted from chondrocytes after amplification, in order to assess whether rotating vessels can be effectively used for cell amplification.

## Specific Aim (3):

Correlate the characteristics of engineered tissues with specific fluid dynamic parameters in order to optimize bioreactor operating conditions.

Rotating bioreactors represent a model system for *in vitro* tissue engineering because hydrodynamic forces can be quantitated and controlled. Viscous coupling induces a steady fluid flow field which entrains inoculated cells, suspends polymer scaffolds, and maintains nascent cell-polymer constructs in a state of continual free-fall. Under these conditions, the relative velocity between the construct and the surrounding fluid can be determined (both numerically and experimentally), used to calculate acting hydrodynamic forces and stresses, and correlated with the biochemical and physical properties of the engineered tissue. In pilot studies, a relative construct-fluid velocity of 2-3 cm/sec and an average hydrodynamic stress of 1.5 dyn/cm<sup>2</sup> promoted chondrocytes to attach to polymer scaffolds and regenerate cartilaginous tissue.

## Collaborations:

1) Extension from chondrocytes to other cell types: Our group at M.I.T. is now working with Dr. Hartzell at duPont to culture cardiac myocytes on PGA scaffolds in rotating vessels. In the pilot study, cell-polymer constructs formed that contracted spontaneously and synchronously at rates of 30-130 beats per min.; such constructs could potentially be used for *in vitro* physiological and pharmacological studies. We also plan to collaborate with Dr. Lelkes at the University of Wisconsin to use other cell types in a similar model system. Optimal culture conditions are expected to depend on the cell type, with respect to mass transfer requirements and shear sensitivity, and on the desired clinical application, with respect to tissue dimensions, structure and function.

2) Extension from simulated to actual microgravity: Flight studies could potentially be done relatively soon using existing NASA hardware, as the first space studies have already been done in parallel to the ongoing ground-based research. Bovine chondrocytes obtained from M.I.T. were flown aboard STS-62 (February, 1994) in an experiment done at the Johnson Space Center. In brief, chondrocytes plated tissue culture dishes were flown in the Biotechnology Specimen Temperature Controller (BSTC) and the cells were cultivated at 37° C for various time intervals (1-4 days). These pilot studies demonstrated chondrocyte growth and metabolism under conditions of actual microgravity.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 3/94 EXPIRATION: 3/95

PROJECT IDENTIFICATION: 962-23-01-11

NASA CONTRACT NO.: NAG-9655

RESPONSIBLE CENTER: JSC

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*Excitable Cells and Growth Factors under Microgravity Conditions*

PRINCIPAL INVESTIGATOR: Dr. Charles R. Hartzell

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**TASK OBJECTIVE:**

Peptide growth factors are intimately involved in the regulation of normal muscle growth and differentiation. Invaluable ground work has been laid by investigators using numerous muscle cell lines to elucidate the contributions made by these growth factors in myogenesis, and significant advances in understanding these mechanistic pathways have been achieved. We underscore, however, the importance of confirming these results in primary muscle cultures.

**TASK DESCRIPTION:**

Using the NASA bioreactor, we will examine the effects of a three-dimensional architecture on the growth and differentiation of neonatal rat heart cells and young adult muscle satellite cells. The bioreactor allows muscle cells to orient and grow within constraints normally determined by the basal lamina *in vivo*, yet permits experimental parameters to be cleanly delineated. Once muscle cultures are established, the role of neuromuscular junction formation on myogenesis will be explored by co-culture of heart or skeletal muscle cells with cholinergic neurons. Alterations in the differentiation program induced by fibroblast growth factor, insulin-like growth factor-I and transforming growth factor- $\beta$  will be evaluated.

**TASK SIGNIFICANCE:**

The two-dimensional, unit-gravity constraints of conventional cell culture do not optimally model the three-dimensional cytoarchitectural design of the *in vivo* system. Limitations inherent in standard cell culture systems encourage us to continue the development of an innovative *in vitro* model system that is not limited by gravity-induced constraints and that promotes the formation of three-dimensional, *in vivo*-like tissue that is critical to understanding myogenic regulation.

**PROGRESS DURING FY 1994:**

1. Neonatal Rat Heart Cells Cultured in HARV Bioreactor

*In vitro* characteristics of cardiac cells cultured in simulated microgravity are reported. Tissue culture methods performed at unit gravity constrain and/or age-dependent cells to propagate, differentiate, and interact in a two-dimensional (2D) plane. Neonatal rat cardiac cells in 2D culture organize predominantly as bundles of cardiomyocytes with the intervening areas filled by non-myocyte cell types. Such cardiac cell cultures respond predictably to the addition of exogenous compounds, and in many ways, they represent an excellent *in vitro* model system. The gravity-induced 2D organization of the cells, however, does not accurately reflect the distribution of cells in the intact heart tissue. The NASA designed High-Aspect-Ratio-Vessel (HARV) bioreactors provide a low shear environment which allows cells to be cultured in static suspension. To evaluate the potential changes induced by the culture conditions of the HARV, neonatal rat heart cells are currently being characterized by a combination of biochemical and cell biological techniques. Since, at the preliminary level, the coordinated activity of the heart depends on specific, sequential events (including cytosolic  $\text{Ca}^{2+}$  elevation, conformational changes and interactions among the contractile proteins, energy availability, and the alignment of myocytes and organization of cellular structures), we are in the process of examining: 1) Contractile Function: cardiac cell beat frequency and strength; 2)  $\text{Ca}^{2+}$  Handling:  $\text{Ca}^{2+}$  distribution, mobilization, and sequestration; 3) Protein Composition: the expression of essential proteins; 4) Energy Metabolism: shifts in metabolism or ATP availability; 5) Cellular Morphology: the

spatial distribution of cardiac cell types; 6) Ultrastructural Morphology: the organization of cellular components and organelles. HARV-3D cultures were prepared on microcarrier beads and compared to control-2D cultures using a combination of microscopic and biochemical techniques. Both systems were uniformly inoculated at  $1 \times 10^6$  cells per milliliter medium per  $4.8 \text{ cm}^2$  surface area (polystyrene). The serum-free defined medium was exchanged at standard 48 hr intervals after day one in culture. Cells in control culture dishes adhered to the polystyrene surface of the tissue culture dishes and exhibited typical 2D organization. Cells cultured in HARVs adhered to the Nunc D-Si polystyrene microcarrier beads, the beads aggregated into defined clusters containing 8 to 15 beads per cluster, and the clusters exhibited distinct 3D layers: myocytes and fibroblasts appeared attached to the surfaces of the beads and were overlaid by an outer cell type. In addition, cultures prepared in HARVs using alternative support matrices also displayed morphological formations not seen in control cultures. Generally, the cells prepared in HARV and control cultures were similar; however, the dramatic alterations in 3D organization gives credence to the generation of tissue-like organizations of cardiac cells in simulated microgravity.

## 2. Rat Skeletal Muscle Satellite Cells Cultured in the HARV Bioreactor

Satellite cells are postnatal myoblasts responsible for providing additional nuclei to growing or regenerating muscle cells. Satellite cells retain the capacity to proliferate and differentiate *in vitro* and, therefore, provide a useful model to study postnatal muscle development. Limiting proliferation and differentiation of satellite cells in 2-D could potentially limit cell-cell contacts important for developing the level of organization in skeletal muscle obtained *in vivo*. Culturing satellite cells on microcarrier beads suspended in the High-Aspect-Ratio-Vessel (HARV) designed by NASA provided a low shear, three-dimensional (3-D) environment to study muscle development. Primary cultures established from anterior tibialis muscles of growing rats (~ 200 gm) were used for all studies and were composed of greater than 75% satellite cells. Different inoculation densities ranging from  $0.5 \times 10^6$  to  $2 \times 10^6$  cells with a cell/bead ratio 20:1 did not affect the proliferation potential of satellite cells in the HARV. Plating efficiency, proliferation, and glucose utilization were determined and compared between 2-D flat culture and 3-D HARV culture. Plating efficiency (cells attached + cells plated  $\times$  100) was similar between the two culture systems and generally was greater than 70%. Proliferation was reduced in 30% of HARV cultures and this reduction was apparent for both satellite cells and non-satellite cells. Furthermore, reduction in proliferation within the HARV could not be attributed to reduced substrate availability since glucose levels in media from HARV and 2-D cell culture were measured to be similar. Morphologically, microcarrier beads within the HARVs were joined together by cells into three-dimensional aggregates composed on 10-15 beads/aggregate. Aggregation of beads did not occur in the absence of cells. Myotubes were often seen on individual beads or spanning the surface of two beads. In summary, proliferation and differentiation of satellite cells on microcarrier beads within the HARV bioreactor results in the formation of a three-dimensional level of organization that provides a suitable model to study postnatal muscle development.

## 3. Myogenic Gene Expression During Postnatal Myogenesis

Rat skeletal satellite cells (postnatal myoblasts) express an intrinsic genetic program that defines them as belonging to the myogenic lineage. This intrinsic program is best exemplified by the ability of satellite cells to differentiate and form contraction-competent myotubes *in vitro*. MyoD, myogenin, MRF4 and myf5 represent a class of muscle-specific transcription factors that modulate the myogenic program during embryonic development, but their role during postnatal myogenesis remains unclear. We have determined the expression pattern for each myogenic factor, and quantitated the level of each factor during different stages of postnatal myogenesis using satellite cells isolated from young adult rats. Reverse transcription polymerase chain reaction (RT-PCR) was used to determine if mRNA for each myogenic factor was present. cDNAs generated by RT-PCR were subcloned and used as specific probes to quantitate the level of each myogenic factor. The following stages of myogenesis were characterized: freshly isolated cells prior to plating (time 0); proliferation (3 day post-plating); day 1 of myotube formation (5 day post-plating), early myotubes (8 day post-plating); late myotubes (13 day post-plating). All myogenic factors were present at each stage examined using RT-PCR. However, levels for each myogenic factor mRNA differed. MyoD level was highest in mononucleated cells. The myogenin level increased 15 fold between day 3 and day 5. MRF4 and myf5 were not detectable until day 1 of myotube formation (using slot blots). In summary, satellite cells isolated from growing rats expressed myogenic factors in a different pattern than what has been reported for embryonic myoblasts and established cell lines.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/92 EXPIRATION: 10/96

PROJECT IDENTIFICATION: 962-23-01-07

NASA CONTRACT No.: NAG-656

RESPONSIBLE CENTER: JSC

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### *Sensitized Lymphocytes for Tumor Therapy Grown in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Marylou Ingram

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#### CO-INVESTIGATORS:

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HMRI  
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#### TASK OBJECTIVE:

The NASA High Aspect Ratio Vessel (HARV) bioreactor is used to generate spheroids of human tumor cells. These spheroids are then used to sensitize the patient's lymphocytes to the patient's own tumor. The population of tumor-sensitized lymphocytes (TSL) is then expanded in culture in the presence of interleukin-2 (IL-2) and used in immunotherapy of the patient.

#### TASK DESCRIPTION:

This is a small scale, in depth pilot study in which nonspecifically stimulated autologous lymphocytes (ASL) and tumor-sensitized lymphocytes (TSL) are administered serially by intralesional injection as local immunotherapy of invasive carcinoma of the urinary bladder. This is a modification of cellular immunotherapy that we have applied with encouraging results in clinical trials to treat recurrent malignant gliomas (1). For the bladder cancer, the urological surgeon will inject cells intralesionally at intervals of approximately three weeks. Response to therapy will be determined by direct observation of the lesions, serial biopsies for histological and cytological studies, clinical evaluations and radiological exams. The ability to biopsy lesions at various times after injection of stimulated immune cells is an important advantage in understanding response to therapy and in refining the therapy. Serial evaluation of lesions by histology, measuring the expression of major oncogenes and tumor suppressor genes, assays of cytotoxic effectiveness of ASL and TSL to tumor obtained from urine specimens or bladder washings can provide additional information between biopsy specimens. The pilot study provides an excellent opportunity to evaluate the NASA HARV bioreactor as a method for culturing tumors as spheroids as contrasted with conventional monolayer cultures. Tumor spheroids *in vitro* have a number of characteristics present *in vivo* but absent from monolayer cell cultures. These characteristics, some of which will undoubtedly be important in the generation of TSL cells, include the presence of specific tumor markers, angiogenesis and growth factors, biological response modifiers, gap junctions and tumor suppressor gene expression. Changes in the level of expression and distribution of intermediate filaments and the extracellular matrix elements have also been observed.

#### TASK SIGNIFICANCE:

Cancer of the urinary bladder is of special interest in immunotherapy because it has already been shown that immunotherapy in the form of BCG treatment is efficacious. It is now widely used in clinical management of invasive bladder cancer. Thus, there is sound evidence that appropriate mobilization of the patient's own cellular immune mechanisms can have therapeutic efficacy. A number of investigators have recognized the possibility that TSL may offer significant advantages in adoptive immunotherapy and there has been a recent resurgence of interest in this approach (2). TSL can be expected to show increased tumor cell killing, as do tumor-infiltrating lymphocytes (TIL) and they also hold forth the promise of serving as probes for identifying immunogenic gene products in the tumor.

Many elderly patients with multiple tumors of the urinary bladder cannot tolerate extensive surgery or extensive chemotherapy and may either have failed to respond to BCG immunotherapy or are too unwell to tolerate side effects of that therapy. These patients present a frustrating therapeutic problem to the urologist. We believe that

local immunotherapy would be well tolerated by these patients and that it is a rational therapeutic strategy. A clinical trial of this therapy will also provide valuable information about how stimulated immune cells select and identify their target cells, migrate through tissue, whether or not they continue to proliferate after injection and other important considerations that will advance our understanding of cellular immunotherapy and aid in refining it for treatment of bladder cancer and ultimately other cancers.

Bladder cancer has many advantages as a model for refining methodology. It permits serial administration of immunologically stimulated cells into the bladder lesions and yields serial biopsy specimens in which the interactions of immune cells and their tumor targets can be studied directly.

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- 2) Greenberg, P.D., Riddell, S.R.J., Natl. Cancer Inst. 84: 1059-61 (1992)

### **PROGRESS DURING FY 1994:**

During FY 94 we have had considerable success in culturing a variety of otherwise "anchorage-dependent" cells lines under conditions of simulated microgravity in the NASA HARV (high aspect ratio vessel) bioreactor. These cells include relatively early passages of some cell lines. To avoid anchorage effects, no microcarrier beads are used. The bioreactor is simply filled with a monodisperse suspension containing 1-2 million cells per ml of nutrient medium. The cultures are maintained for periods of a few days to approximately a month. The spheroids produced are fixed in Carnoy's/Bouin's fixative, then sectioned and stained. Some sections of each specimen are stained with hematoxyline and eosin and other sections from the same specimen are stained immunohistochemically to demonstrate various markers of interest.

When cultured in the bioreactor, each cell line produces spheroids that demonstrate cell line-specific microscopic architecture. Thus, gliomas tend to form small, compact spheroids that sometimes fuse to form larger bodies; the PC3 cell line that was developed from prostate carcinoma metastatic in bone forms discrete spheroids in which cells are very loosely associated. Another prostatic carcinoma cell line, LNCaP, typically forms elongated tubule-line or folded structures. The bladder carcinoma cell line, HBL2, recently developed here, shows a definite tendency towards epithelial differentiation. The tendency for the HBL2 cells to assemble in such a pattern suggests that various adhesion molecules play a role in determining spheroid morphology. Immunohistochemical staining to demonstrate fibronectin, collagen, vimentin, CD44 and other cell adhesion molecules can be expected to provide further insight into spheroid formation and differentiation. These studies are in progress.

An abstract and poster describing results of cell culture in the NASA bioreactor were accepted for presentation in the "Hot Topic" poster session at the Congress on Cell and Tissue Culture Regulation, Cell and Tissue Differentiation convened by the Tissue Culture Association, June 4-7, 1994 at Research Triangle Park, N.C. Dr. Ramez Saroufeem, who had primary responsibility for the bioreactor cultures of the HBL2 cancer cell line, attended the meeting and presented the poster.

Our clinical collaborator, Dr. Michael Bishai, urological surgeon, has provided fifteen surgical specimens of bladder tumors and these have been invaluable. They have allowed us to refine tissue culture methods specifically for bladder cancer. Most of the patients from whom the specimens were obtained did not meet protocol requirements (patient selection criteria) for the immunotherapy trial and Dr. Bishai managed these patients clinically using other therapeutic modalities. The fifteenth patient does meet protocol requirements and has just been entered as the first protocol patient.

From the bladder cancer specimens obtained so far, we have established one bladder cancer cell line and, at least one additional specimen may also yield a permanent cell line. The new cell line, designated HBL2, has been cultured in both monolayer culture and in the NASA bioreactor culture vessel. HBL2 cells, as well as minced tumor from some of the other bladder cancers and glioma and prostate cancer cell lines, provide important experimental and reference specimens for immunohistochemical demonstration of "markers" of interest and for comparison of "marker" expression in cells grown in monolayer as contrasted with the same cell lines grown as spheroids or examined in

tumor mice specimens. Although we cannot, of course, expect to establish a permanent cell line (i.e., at least 25 passages) from every tumor, primary cultures and tumor mince will be available for use in preparing TSL in all cases. Where possible, lymphocytes to be tumor-sensitized for bladder cancer immunotherapy will be exposed to tumor mince, monolayer cultures, and tumor spheroids in parallel so that we may compare the relative immunological effectiveness of the three modes of tumor presentation. We also hope to establish, from a least one tumor, a line of fibroblasts and a line of endothelial cells as well as tumor cells and to use these autologous cells in a structured series of co-cultures to observe histogenesis and the effects of active histogenesis on the expression of antigenic gene products.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/92    EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-23-01-14

NASA CONTRACT No.: NAG-649

RESPONSIBLE CENTER: JSC

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*Three-Dimensional Modeling of Human Colon Tissues*

PRINCIPAL INVESTIGATOR: Dr. J. M. Jessup

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CO-INVESTIGATORS:

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**TASK OBJECTIVE:**

The objectives of this project are to determine whether 1) microgravity permits unique, three-dimensional cultures of neoplastic human colon tissue and 2) this culture interaction produces novel intestinal growth and differentiation factors.

**TASK DESCRIPTION:**

The initial phase of this project will test the feasibility of microgravity for the cultivation and differentiation of human colon carcinoma. We propose to do this in rotating wall vessels (RWVs) which provide a low shear stress environment in unit gravity. In this environment, early experiments have demonstrated normal human colon fibroblasts stimulate the differentiation of certain human colon carcinoma cell lines so that they produce three-dimensional tissue masses that are similar to neoplasms in patients or in xenografts in athymic nude mice.

**TASK SIGNIFICANCE:**

The important question is whether this differentiation induced by fibroblasts is due to the low shear stress environment of the RWV or whether suspension cultures in the RWVs are similar to cultures in standard culture systems in unit gravity. Should the low shear stress environment of the RWV be superior to that of conventional culture systems, then the co-culture experiments should be attempted in an actual microgravity environment.

**PROGRESS DURING FY 1994:**

This grant was completed in May 1994, and its purpose was to determine whether normal stromal cells coated with epithelial cells would form three-dimensional structures that were similar to tissues that developed in mice or humans. We studied adenocarcinomas of the colon and began studies with normal colonic epithelium. We did not culture normal colonic epithelium in the rotating wall vessel (RWV) because we were not able to obtain a small (50ml) RWV. Nonetheless, we did demonstrate that normal colonic epithelial cells isolated from colonic crypts adhered to collagen Type IV and to carcinoembryonic antigen (CEA)-coated surfaces. This suggests that CEA is an intercellular adhesion molecule that may be used by human normal colonic epithelial cells, while normal colonocytes use collagen Type IV as a ligand in their basement membrane. This work resulted in a publication in *Gastroenterology*. As indicated in that publication, normal colonocytes do not survive more than 24-48 hours when cultured on either CEA or collagen Type IV. Their ability to remain viable is better on collagen Type IV than on any other substrate including laminin and fibronectin-coated surfaces. However, because survival is so short for these isolated colonocytes, we did not feel that we could put these cells into the RWV unless they were cultured in small volumes so that we could use sufficient spent medium to try to prolong their survival.

We have attempted to isolate normal human stromal cells from colonic surgical specimens. However, the amount of tissue that we have obtained from our pathology department has been too small to initiate primary cultures of colonic fibroblasts in sufficient quantity to allow stromal cell cultures. As a result, we attempted to use a mouse embryonic fibroblast cell line, BALB/c 3T3 cells as the stromal feeder cell. When we did this, the 3T3;MIP-101 cells did not interact well and did not grow together in suspension. The MIP-101 cells actually shunned the 3T3-coated microcarrier beads and the MIP-101 colon carcinoma cells grew in suspension.

Our rationale for this experiment is that these human colonic adenocarcinoma cell lines develop different degrees of differentiation when grown as xenografts in mice and this requires interaction with mouse stromal cells. We also reasoned that an immortal embryonic fibroblast line may mimic colonic fibroblast function under appropriate in

*vitro* conditions. We did not repeat this with the other cell lines because they will behave much like HT-29. We did culture HT-29 KM with 3T3 cells and found that they behave similarly to the human colonic fibroblast: HT-29 KM co-cultures.

This study was to investigate the production of novel intestinal growth factors. We had initially developed a cumbersome bioassay in which potential factors were encapsulated in liposomes and injected into the cecal wall of mice. Growth and differentiation of mucin-filled goblet cells was evaluated by morphological assay of colonic crypts overlying the liposomal implant. This assay was extremely operator-dependent and was not easily reproduced in Boston. As a result, we attempted to develop a series of *in vitro* bioassays which examined the amount of antigen expressed on the cell surface by a cell based enzyme immunoassay. The difficulty with this type of assay is that none of the six colorectal cell lines that we examined had stable expression of the mucin related epitopes that we were evaluating. Thus, the ability to demonstrate the goblet cell differentiating factor (GCDF) activity that had initially been defined in the *in vivo* bioassay was not supported by a subsequent *in vitro* assay. Nonetheless, we did evaluate the expression of EGF, bFGF, and TGF- $\beta$  1 were released by these colon carcinoma cell lines into the surrounding medium. Interestingly, recent data from other laboratories suggest that well or moderately differentiated carcinomas are inhibited by TGF- $\beta$  1, whereas poorly differentiated cell lines may be stimulated by TGF- $\beta$  1. Our results indicate that LS-174T cultures plateau as the TGF- $\beta$  1 concentration increases. However, the other three colon carcinoma cell lines did not demonstrate a consistent relationship among the proliferation, viability and TGF- $\beta$  1 level. MIP-101 cultures, for instance, demonstrate a decrease in TGF- $\beta$  1 concentrations even as they enter a plateau phase.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 4/91 EXPIRATION: 4/94

PROJECT IDENTIFICATION: 962-23-01-05

NASA CONTRACT NO.: NAG-520

RESPONSIBLE CENTER: JSC

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### *Three-Dimensional Tissue Interactions in Colorectal Cancer Metastasis*

PRINCIPAL INVESTIGATOR: Dr. J. M. Jessup

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### **TASK OBJECTIVE:**

The objective of this project is to test the fidelity with which microgravity models three-dimensional tissues by assessing how well microgravity effects the molecular and biological function of MIP-101, a human colorectal carcinoma.

#### **TASK DESCRIPTION:**

MIP-101 is a poorly differentiated adenocarcinoma of the colon that was derived from the ascites of a patient who had widespread metastases within the abdominal cavity. MIP-101 cells are grown in the rotating wall vessel (RWV) with or without normal host cells and assessed for the production of carcinoembryonic antigen (CEA - a 180 kDa glycoprotein that is produced by carcinomas and used clinically as a tumor marker) and for biological behavior including adhesion and metastasis. Cells grown in microcarrier bead cultures in the RWV under simulated microgravity are compared to similar MIP-101 cultures grown on microcarrier beads in stationary culture as well as in conventional monolayer cultures.

#### **TASK SIGNIFICANCE:**

One of the prime goals of the biotechnology program at NASA/Johnson Space Center is to determine whether cultivation of cells in microgravity produces three-dimensional cultures that mimic the morphology and function of tissues in living animals or humans. The MIP-101 cells are an excellent test of this because they are poorly metastatic in experimental models of metastasis in athymic nude mice and do not produce CEA in conventional monolayer culture systems. We have shown that CEA injected into mice enhances production of liver metastases by MIP-101 cells and that MIP-101 cells will metastasize when implanted into the abdominal cavity after producing CEA. MIP-101 cells placed in the subcutaneous tissue of the mouse do not metastasize and do not produce CEA. Furthermore, cells grown on plastic or conventional substrates such as Matrigel or laminin are neither metastatic nor induced to produce CEA.

The conventional interpretation of these results is that the microenvironment (the three-dimensional environment) of the abdominal cavity induces MIP-101 cells first to produce CEA and then to develop blood-borne metastases in the liver and lungs of nude mice. Early experiments in the RWV question this conventional interpretation of the effects of host microenvironment, because they demonstrated that CEA production may be induced in MIP-101 cells when they are grown in the RWV in the absence of any abdominal cavity stromal cells. This suggests that the MIP-101 cells may produce CEA when they are allowed to grow in three dimensions. Thus, the absence of CEA production in subcutaneous tumors is due to an inhibition of CEA production by the subcutaneous tissue rather than promotion of CEA production and metastatic potential by the abdominal cavity.

This system is an excellent model in which to test the fidelity of the RWV culture system because the first stage in metastasis by MIP-101 cells appears to be the induction of CEA production, followed by the acquisition of the ability to develop experimental metastases after injection into the spleen of nude mice. Assessment of fidelity of the RWV culture system is simplified by first testing for the production of CEA, tumor marker, then assessing the biological aspects of metastasis.

### PROGRESS DURING FY 1994:

The primary goal of the Biotechnology Program at NASA/Johnson Space Center is to determine whether cells cultivated in microgravity produce three-dimensional cultures that mimic the morphology and function of tissues in living animals or humans. The MIP-101 cells are an excellent test of this because they are poorly metastatic in experimental models of metastasis in athymic nude mice and do not produce CEA in conventional monolayer culture systems. We have shown that CEA injected into mice enhances production of liver metastases by MIP-101 cells and that MIP-101 cells will metastasize when implanted into the abdominal cavity as they produce CEA. Recently, a CEA-producing transfectant of MIP-101 cells was found to be metastatic in nude mice. However, parenteral MIP-101 cells placed in the subcutaneous tissue of the mouse do not metastasize and do not produce CEA. Furthermore, cells grown in plastic or conventional substrates such as Matrigel or laminin are neither metastatic nor produce CEA at detectable levels by either RT-PCR or western blots.

Since the three-dimensional growth in the peritoneal cavity induces the production of CEA, it was thought that peritoneal stromal cells were able to induce CEA production by MIP-101 cells. Interestingly, when MIP-101 cells were grown as three-dimensional colonies in the rotating wall vessel (RWV), CEA was released into the medium, and immunoperoxidase staining of MIP-101 cultures revealed that MIP-101 cells produced CEA in the majority of CEA cells forming three-dimensional aggregates. Thus, it appears that production of CEA by MIP-101 cells is enhanced by three-dimensional growth as opposed to two-dimensional growth in monolayers.

Recently, more data has been obtained to support this interpretation. Interestingly, some experiments have shown that growth in Petri dishes or in T-25 flasks leads to CEA production as determined by western blot analysis as well as by enzyme immunoassay of the spent culture medium. RT-PCR demonstrates gene transcripts in these conventional culture systems when MIP-101 cells have been cultivated for more than 8 days. The RT-PCR, however, shows that the gene transcripts are still quite low in number and that, if RT-PCR is performed under stringent conditions, the quantity of mRNA for CEA is below the limits of detection. In contrast, RT-PCR and western blots clearly demonstrate the presence of the 180 kDa protein in RWV cultures of MIP-101 as early as 4 days after the initiation of RWV cultures. In addition, CEA proteins are identified by immunoperoxidase staining at day 4 as well as by western blots of extracts of MIP-101 cells beginning at day 8. The findings of low CEA production by MIP-101 cells in Petri dishes and T-25 flasks was somewhat unexpected, but was found to be associated with overcrowding and "piling up" of MIP-101 cells as the cultures grew vertically. Thus, in the late phases of the MIP-101 cultures, since they are not contact-inhibited, the cells assume a three-dimensional formation that is somewhat similar to the aggregates that are formed in the RWV. Thus, it is clear that there is a very low amount of CEA produced in MIP-101 cells which can be dramatically increased as the cells enter a three-dimensional culture formation.

The factors that regulate the three-dimensional formation and how this alters cell growth are being investigated. It is clear that, in the three-dimensional aggregates formed in the RWV, the metabolic rate of the MIP-101 cells is dramatically increased. A comparison of the MIP-101 cells grown in zero head-space T-25 flasks with the RWV at similar cell concentrations indicates that far more  $H^+$  ion and  $CO_2$  are produced in the RWV than in the monolayer culture. However, the amount of glucose utilized is not significantly different. This suggests that an alternative fuel source is being used by these cells in the RWV that leads to the production of acid metabolites. Future experiments are designed to determine how these different metabolic pathways are activated because they may impact on the regulation of CEA production.

In addition, studies are underway to identify how the peritoneal cavity improves the metastatic potential of these MIP cells and, notwithstanding the effects of MIP cells grown in isolation on CEA production, there are still stromal cell:MIP cell interactions. Recent work with Dr. Catherine Chen, who is doing a research fellowship in Dr. Judah Folkman's laboratory, indicates that MIP-101 cells grow preferentially along the greater curvature of the stomach of nude mice. Dr. Chen has found that most human neoplasms prefer to grow in this part of the peritoneal cavity as compared to the mesentery or other peritoneal surfaces in the abdominal cavity. If this is true, then it should be possible to isolate the cells that support this growth and use them as stromal cells in future RWV co-cultures with MIP-101 cells. We plan to do this in the next part of the fiscal year as well as to investigate the metabolic pathways that are involved with induction of CEA production by MIP-101.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-01-13

NASA CONTRACT NO.: NAG-650

RESPONSIBLE CENTER: JSC

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*Applications of Atomic Force Microscopy to Investigate Mechanisms of Protein Crystal Growth*

PRINCIPAL INVESTIGATOR: Dr. John H. Konnert

Naval Research Laboratory (NRL)

CO-INVESTIGATORS:

K. Ward

Naval Research Laboratory (NRL)

P. D'Antonio

Naval Research Laboratory (NRL)

**TASK OBJECTIVE:**

The research objective is to use Atomic Force Microscopy to study protein crystal growth mechanisms by extending the pioneering work of Durbin et al (1992) to include crystals other than lysozyme, and by applying image analysis methods not generally available to others to aid in the interpretation of crystal face images observed by this technique. Specific objectives include:

- To modify an AFM liquid sample cell for use as a crystal growth cell for protein crystals. This cell has a flow-through system which will allow protein supersaturation to be controlled by varying the temperature of the crystallization solution bathing the crystal being observed;
- To determine by direct observation of developing crystal faces the mechanism of crystal face growth for a variety of growth conditions including solution composition, degree of protein supersaturation, temperature, growth rate, and microgravity;
- To use crystal etch figures methods and direct observation of growing crystal faces observed by AFM to classify and determine the number of crystal imperfections which occur under a variety of crystal growth rates and conditions, including the examination of faces of single crystals prepared under microgravity conditions;
- To demonstrate that the observed diffraction quality of protein crystals prepared under a variety of conditions can be correlated with the number and type of crystal defects observed using AFM; and
- To determine whether any observed changes in the appearance of the surface of protein crystals can be correlated with the growth cessation phenomena of protein crystals.

**TASK DESCRIPTION:**

Proteins will be prepared for examination by AFM using conventional vapor diffusion, hanging drop methods, and in the temperature-controlled crystallization cell described by Ward, Perozzo and Zuk (1992).

The goal will be to prepare crystals of a given protein using different growth rates by carefully controlling the degree of supersaturation and other growth parameters. Single crystals prepared for these studies will be characterized by x-ray diffraction analyses using standard data reduction and analysis techniques. The diffraction quality will be quantified using the relative Wilson plot analyses described by DeLucas et al (1991).

In addition to preparing crystals under unit gravity conditions, we also intend to submit these proteins for crystallization experiments under microgravity conditions. These experiments will be performed by Keith Ward either as part of the co-investigator protein crystallization program at the University of Alabama-Birmingham or as part of his own Flight Investigation Project which has been submitted in response to the recent NRA.

A number of proteins have been selected for this application of AFM to protein crystal growth studies. Each one is readily available, easily crystallized, and exhibits unique crystallization properties.

**TASK SIGNIFICANCE:**

This project will, for the first time, provide unique information about protein crystal growth processes by direct observation of crystal faces in their growth medium. Successful results will provide new evidence for the effect of crystal growth conditions, including microgravity, on the defect structures and diffraction quality of protein crystals. Results derived from this project will, therefore, be of direct significance to NASA-funded efforts aimed at preparing high-quality protein crystals for structural investigations by effectively utilizing the unique microgravity environment of space platforms.

**PROGRESS DURING FY 1994:**

Techniques have been developed to directly observe with the AFM the growth kinetics of protein crystals in solution whose concentrations span those employed for the growth of crystals suitable for single crystal diffraction experiments. These observations at the molecular level permit the macroscopically observed crystal growth rates to be factored into two components:

1. The growth of existing features as a function of crystallographic orientations.
2. The nucleation of new features on the existing surface as a function of crystallographic orientation. In addition, our system allows both components to be investigated as a function of composition and temperature of the solutions from which the crystals grow. A journal article is being prepared.

During the next fiscal year, we will extend our studies to other protein crystals, in order to test the general applicability of this method for discerning details about protein crystal growth mechanisms.

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**STUDENTS FUNDED UNDER RESEARCH:****TASK INITIATION:** 1/93 **EXPIRATION:** 1/96**PROJECT IDENTIFICATION:** 962-23-08-25**NASA CONTRACT No.:** H-07973D**RESPONSIBLE CENTER:** MSFC

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*Neuro-endocrine Organoid Assembly in Vitro*

PRINCIPAL INVESTIGATOR: Dr. Peter I. Lelkes

University of Wisconsin, Milwaukee

CO-INVESTIGATORS:

B.R. Unsworth

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**TASK OBJECTIVE:**

The specific aims of our project, entitled "Neuro-endocrine Organoid Assembly In vitro" are as follows:

1. To assemble adrenal medullary endothelial and parenchymal cells into functional organoids. Progress will be monitored by evaluating, a) morphology (light microscopy and ultrastructure), b) intercellular communication (immunocytochemistry), c) functional maturation and its hormonal control by corticosteroids, d) the expression of phenotypic, biochemical, and molecular markers.
- 2) To compare the usefulness and efficacy of NASA vessels with conventional co-culture systems (monolayer culture, suspension culture and 3-dimensional gel assembly). The time course, and the extent of neuro-endocrine cell differentiation, under the different culture conditions will be evaluated (as in 1).

**TASK DESCRIPTION:**

Our long term research goal is to understand the fundamental mechanisms of neuro-endocrine gland assembly and differentiation. In our particular model system, the adrenal medulla, neural crest-derived cells of the sympathoadrenal lineage differentiate into neuro-endocrine chromaffin cells. We are particularly interested in the role of the microenvironment in this process. We have previously shown that during assembly and maturation of the adrenal medulla, as in other endocrine organs, parenchymal cells and endothelial cells interact through reciprocal intercellular signals. Such signals may constitute soluble factors, heterotypic cell contacts, or may be derived from organ-specific extracellular matrix components. These cues comprise part of the epigenetic repertoire, which induces the ordered differentiation of both cell types into what is known as the "endocrine structure".

Using conventional 2-D culture techniques, we have shown that in co-culture with adrenomedullary endothelial cells, chromaffin-cell-derived pluripotent PC12 cells differentiate towards the neuroendocrine phenotype. We, therefore, hypothesize that capillary endothelial cells in the adrenal medulla provide some of the organ-specific, differentiative cues which contribute to the neuroendocrine differentiation of the chromaffin precursor cells.

In this project we extend our on-going *in vitro* studies on the mechanisms of organ-specific differentiation by using alternate methods of co-culture. We are exploiting the enhanced potential offered by the NASA vessels to analyze the temporal assembly of co-cultured adrenomedullary endothelial and parenchymal cells into functional, organellal-like structures (organoids). The simulated microgravity environment of the NASA vessels has been shown to randomize gravitational vectors and minimize detrimental shear forces routinely encountered in conventional three-dimensional suspension cultures in spinner flasks. We anticipate that the favorable culture conditions in the NASA vessel will accelerate differentiative, heterotypic cell-cell contacts and thus lead to differentiated organoid-assembly *in vitro*.

**TASK SIGNIFICANCE:**

In this study, we attempt, for the first time, to generate neuro-endocrine organoids *in vitro* by co-culturing different cells isolated from the same organ under simulated microgravity conditions. By using the NASA cell culture vessels as a novel, alternate approach to conventional suspension culture in spinner flasks, we are participating in NASA's assessment of simulated microgravity conditions for organ-specific culture and cellular differentiation. As previously shown, a major advantage of using the Rotating Wall Cell Culture Vessels (RWVs) developed by NASA is the enhanced cell viability and tissue differentiation under conditions of simulated microgravity and minimized shear stress.



The unique conditions in the NASA vessels are believed to enhance cellular interactions and thus differentially accelerate and/or facilitate these processes. In complementing a number of other parallel studies within this program, our project is the first one specifically designed to examine the effects of microgravity on heterotypic interactions between parenchymal cells and microvascular endothelial cells isolated from the same organ, namely the rat adrenal medulla. Such intercellular interactions are believed to be of importance during all phases of the development of endocrine organs, from the earliest stages of embryonic genesis through postnatal maturation.

By using NASA's RWVs as the prime cell culture environment, we will be able to assess whether the spatial arrangement of the functional organ is affected by gravitational forces/vectors. Based on our preliminary observations, we hypothesize that by using the RWVs, we will be able to eliminate gravitational vectors, such as those present in static two-dimensional monolayers or in suspension cultures, and thus obtain a more realistic representation of tissue assembly, as seen *in vivo*.

Our co-culture system is ideally suited for dissecting the microenvironmental effects (heterotypic cell interactions, microgravity, etc.) on neuro-endocrine differentiation of adrenal medullary chromaffin cells, since many of the phenotypic and genotypic markers for organ specific differentiation of these cells are well characterized. Furthermore, our model system of heterotypic co-culture of adrenal medullary cells is of particular relevance in view of our recent finding that microgravity specifically alters the expression of pivotal enzymes in the catecholamine synthesizing cascade. Thus, our model system is also suitable to explore the cellular and molecular basis for (micro)gravity sensitivity, *e.g.*, of signal transduction mechanisms involved in neuroendocrine hormone synthesis and secretion.

Based on the first year of practical experience with one of the RWVs, we are confident that the enhanced cellular viability and differentiation achieved by culturing the cells under similar microgravity conditions will be of general advantage for developing new concepts for tissue culture and tissue engineering. Specifically, we anticipate that this novel environment will be beneficial for culturing and/or co-culturing fragile cell types, *e.g.*, when isolated at early stages of embryonic development. We anticipate that within a reasonable time period, we will be able to test our model for organogenesis and differentiation during one of the future Space Shuttle missions.

#### PROGRESS DURING FY 1994:

1. As indicated in last year's progress report, we spent the first year of the grant on refining our basic heterotypic cell culture systems using conventional monolayer and suspension cultures. In collaboration with scientists at JSC, (specifically the group of T. Goodwin) we explored the suitability of the various available rotating-wall vessels (RWVs) for growing adrenal medullary parenchymal and endothelial cells, alone and in co-culture, under simulated microgravity conditions. Based on those preliminary findings, we decided to continue our studies in the Slow Turning Lateral Vessel (STLV) type RWV.
2. After in-servicing a graduate (Ph.D.) student, Daniel Galvan, at JSC, two STLVs were shipped to Milwaukee in the beginning of 1994. We are currently carrying out the experiments according to the plans detailed in the specific aims; we are assessing the effects of simulated microgravity on neuro-endocrine organ-assembly by co-culturing transformed rat adrenal medullary parenchymal cells (PC12 pheochromocytoma cells) with organ-specific microvascular endothelial cells (RAME, rat adrenal medullary endothelial cells). In spite of some technical difficulties with the device drive units, the prolonged duration of each of these experiments (up to 3 weeks), and the limited number of vessels available (two), we have obtained some encouraging preliminary results:
  - a) We optimized the conditions for culturing each of the cell types alone and in co-culture on microcarrier beads in the STLV. The efficiency of cell culture was assessed, *e.g.*, by the rates of glucose utilization rate and proliferation, respectively.
  - b) Preliminary visualization by routine histological staining of the ensuing three-dimensional cell aggregates suggests that both PC12 and RAME cells form histotypic assemblies on the beads; large clumps/aggregates of viable PC12 cells reminiscent of parenchymal acini and contact-inhibited monolayers of RAME cells. By contrast, in the co-cultures, we observed organotypic assemblies of mixed populations, in which homotypic interactions prevail. Thus, PC12 remain clustered into nest-like assemblies, surrounding (or surrounded by) RAMEs.

Importantly, for the first time, we also observed in these co-cultures, the assembly of RAMEs into what appear to be capillary structures. These experiments are currently being repeated to establish the reproducibility of the exciting observation that co-culture, under simulated microgravity, results in organotypic assembly of adrenal medullary cells and enhances organ-specific differentiation of both parenchymal and endothelial cells. Currently, the findings are being substantiated by molecular biological and immunochemical techniques of the light and electron microscopic level. As described below, we implemented a novel quantitative PCR technique for measuring the expression of genes related to neuroendocrine differentiation. In addition, studies are under way to assess, by immunohistochemical means, whether the seemingly histotypic assembly of neuro-endocrine "organoids" in the STLV is accompanied by the enhanced expression of organ-specific differentiation markers, such as the catecholamine synthesizing enzymes in adrenomedullary PC12 cells.

c) We previously reported that PC12 cells and RAME formed organ-specific assemblies in conventional suspension cultures which were maintained at low speed with minimal shear forces in the absence of microcarrier beads. In repeating the experiments, we now have substantiated these findings. Specifically, RAME cells were found to attain configurations which are reminiscent of the formation of new blood vessels (angiogenesis). Since, in the STLV cells cannot be cultured without beads, we have begun to optimize conditions for culturing the cells in a small (10 ml) High Aspect Ratio Vessel (HARV). These experiments are designed to permit direct comparison between different "beadless" cell cultures in stirred fermentors vs. in a RWV. We expect that this comparison will yield relevant information on the beneficial effects of simulated microgravity on organ-specific differentiation.

3. As detailed above, the focus of our studies is to assess differential expression of organ-specific marker genes by using molecular biological techniques. In the past year, we have made significant progress in several complementary areas:

a) We continue to assess the effects of simulated microgravity on differential gene expression, mainly via RT-PCR. To increase the reliability of our results and to eliminate the possibility of false positives, we have synthesized, verified, and tested a number of previously not available species (rat)-specific PCR primer pairs for the following rat-specific genes:

Comparator genes for relative quantitation: glyceraldehyde-phosphate-dehydrogenase (GAPDH),  $\beta$ -actin and (mitochondrial) rRNA

Catecholamine synthesizing enzymes: tyrosine hydroxylase (TH), phenylethanolamine-N-methyltransferase (PNMT), dopamine- $\beta$ -hydroxylase (DBH), aromatic-dopa-decarboxylase (DDC)

Protooncogenes: *c-fos*

Catecholamine metabolizing enzymes: monoamine-oxidase A (MAO-A), and B (MAO-B)

Extracellular matrix proteins: fibronectin (FN). Currently, rat-specific PCR primer pairs and RNA/DNA probes are being established for other extracellular matrix proteins, such as collagens I and IV, laminin A and B1 chains, for which so far only the murine sequences are known.

b) Having established species-specific PCR primer pairs and probes, we are currently constructing species-specific sense and antisense RNA probes for Northern blotting, RNase protection assays and for *in situ* hybridization studies. These techniques will complement our quantitative PCR approach (see below).

4. In the past, we have semi-quantitated differential gene expression by RT-PCR using established comparator genes, such as GAPDH,  $\beta$ -actin or rRNA (Lelkes, et. al., FASEB J. in press, see appendix). Recently, we established a rigorous, quantitative PCR-assay, termed competitive RT-PCR. In this assay, a so-called PCR mimic DNA template is added to the reaction buffer prior to the PCR step. This mimic uses the same primers as the endogenous mRNA sequence, but slightly differs in size, which allows its separation from the endogenous one on the DNA gel. Therefore, mimic templates compete with endogenous cDNA for PCR substrates. By adding a known number of mimic templates equal to the number of endogenous templates into the PCR reaction, two products are obtained of equal intensity and slightly different sizes as visualized on a DNA electrophoresis gel. To date, we have generated and successfully tested PCR mimics for TH, PMNT and FN (see below).

5. As detailed above, our long term goal is to study the molecular mechanisms by which microgravity might affect cellular differentiation. Specifically, we want to discover novel molecule(s) involved in the microgravity-induced neuroendocrine differentiation. Towards that goal, we have in the last year implemented the most recently developed molecular technique, called Differential Display. Using this technique, the RNA populations of both microgravity-treated and control samples can be displayed on a 2-D sequencing gel. The identities of any known or unknown RNAs which are differentially expressed in the PC12 cell culture in microgravity vessel can be revealed by

further PCR, cloning and sequencing. So far, we have successfully tested this technique and optimized the assay conditions. Current experiments are under way to employ the differential display assay for studying the genes which are differentially activated in the PC12 cells exposed to microgravity.

6. Other related studies:

a) As described in last year's report, we used some of the biochemical and molecular techniques to assess the effects of microgravity on the expression of catecholamine synthesizing enzymes in the adrenal medulla of rats flown aboard the Space Shuttle Mission STS-54. In addition to presenting these results at the annual meeting of the American Society of Cell Biology (Lelkes, et al., Mol. Boil. Cell, vol 4 Supply:109a, see appendix), a paper entitled "*Microgravity Decreases Tyrosine Hydroxylase Expression in Rat Adrenals*" is currently in press in the FASEB Journal (see appendix). In brief, our data show that the expression and specific activity of tyrosine hydroxylase (TH), the rate limiting step in catecholamine synthesis was specifically affected by microgravity. By contrast, phenylethanolamine-N-methyl-Transferase (PNMT), the chromaffin cell-specific enzyme which converts norepinephrine to epinephrine was not affected during space flight. We hypothesized that these observed effects of space flight were not due to stress response during reentry because of the relatively long time course (about 12 hours) of TH depression and lowered catecholamine contents. Furthermore, based on evidence in the literature, we speculated that microgravity may be exerting its effect on catecholamine synthesis by acting on specific, microgravity-sensitive signal transduction pathways.

We have now further substantiated these conclusions by analyzing adrenals of (10 days) tail-suspended rats, a putative, ground-based control for weightlessness. For these studies, we used for the first time our newly developed method of competitive PCR to quantitate TH and PNMT expression. In contrast to the pattern of TH and PNMT expression in the adrenals of space-flown animals, we found that in the adrenals of tail-suspended rats, the expression (and specific activities) of both TH and PNMT were increased by, respectively, 30% and 100%. This pattern of elevated catecholamine synthesizing enzyme levels is typical for stress situations, suggesting that prolonged tail suspension, while presumably suited for studying the effects of simulated weightlessness on muscle physiology, might not be suited to model any effects of microgravity on the (neuro) endocrine system.

b) In order to simplify cell culture in the RWVs, the biotechnology group at JSC recently developed a novel cell culture medium called GTSF-2. In collaboration with the scientists in that group, we completed our evaluation of GTSF-2 using a number of different cell types and other with our cells. A paper, entitled "*GTSF: A New, Versatile Cell Culture Medium for Diverse Normal and Transformed Mammalian Cells*" is currently in press in the Journal *In Vitro*. Our data suggest that GTSF-2, by itself or with minor modifications, is well suited to maintain the growth of a variety of cell types including neuronal cells, primary cultures of endothelial cells from humans and rodents, and several lines of transformed cells.

c) An exciting spinoff of our ongoing studies is the use of RWVs to culture normal proximal tubular epithelial cells isolated from human kidneys. These cells are notoriously difficult to handle and very rapidly de-differentiate in conventional 2-D cultures. In collaboration with Dr. T. Hammond (U. Wisc. Med. School) and Clonetics Co. (San Diego), we have used the simulated microgravity in an STLV to establish both morphologically and functionally highly differentiated cultures of these cells. An abstract (*Human Renal Epithelial Cells in Culture Differentiate Under Simulated Microgravity*) will be presented at the upcoming Annual Meeting of the American Society for Cell Biology. A manuscript detailing our results is in preparation. Moreover, the budding collaboration with a small business company raises the prospect that, besides their usefulness as novel research tools, RWVs can be used for developing a commercializable product which will be important for basic and applied biomedical studies.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 11/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-23-01-12

NASA CONTRACT NO.: NAG9-651

RESPONSIBLE CENTER: JSC

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*Formation of Ordered Arrays of Proteins on Surfaces*

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PRINCIPAL INVESTIGATOR: Prof. Abraham M. Lenhoff

University of Delaware

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The goal of the project is to investigate the formation of ordered arrays of proteins at solid surfaces. Two parallel approaches are employed:

1. An empirical study, by scanning probe microscopy (SPM), of the effects of protein and surface parameters on array formation;
2. A fundamental examination, by molecular simulations and surface forces apparatus measurements, of the protein - protein and protein - surface interactions that give rise to protein adsorption and ordering.

Although our primary interest is in proteins, insight can be provided in both the simulations and experiments by corresponding experiments on spheres. For this purpose, the FY94 task included study of amidinated polystyrene latex particles.

**TASK DESCRIPTION:**

The tasks begun in FY94 fall into three categories:

1. Atomic force microscopy (AFM) of proteins and latex spheres on surfaces, complementing the purely protein-related work performed and proposed previously. Use of AFM makes possible the investigation of behavior at non-conducting surfaces, specifically mica, while examination of latex spheres makes possible discrimination among multiple possible explanations for changes in adsorption extent as a function of salt strength.
2. Calculation of the energetics and hence the isotherms for adsorption of ordered arrays of particles at charged surfaces. Our computations to date on ordered arrays have examined only spherically symmetric particles, and the additional studies are intended to investigate the effects of charge asymmetry and possibly of particle shape.
3. Surface forces measurements of protein - surface interactions. Experimental measurements of the interaction energy of ordered arrays of streptavidin, immobilized on one surface, with a second charged surface.

**TASK SIGNIFICANCE:**

Ordered arrays of proteins are of interest for two main reasons:

1. Protein structure determination. X-ray crystallography is still the workhorse for determination of protein 3-D structures, but other techniques have begun to emerge. One class of methods that emerged during the 1980s is that of structure determination on two-dimensional crystals, with techniques such as electron microscopy employed to probe the structures of the constituent molecules. The 2-D arrays we will study would be suitable for such investigations. Furthermore, it is possible that such arrays can serve as templates for the epitaxial growth of 3-D crystals.
2. Synthesis of novel materials. The complex 3-D structures of proteins give them functional properties that are also complex, and in addition can be produced reproducibly. Given proteins with the appropriate functions, ordered monolayers of protein molecules at solid surfaces may represent materials with useful properties that make various applications possible. Examples include electronic devices and "biocomposite" materials.

Apart from these applications, our studies of self-assembly have a further, more fundamental purpose: the colloidal forces driving 2-D self-assembly are the same as those giving rise to 3-D crystal growth, so understanding these forces and their interactions is expected to lead ultimately to insights into manipulation of both 2-D and 3-D structures. It is also the isolation of these colloidal forces from other disturbances that provides the incentive for microgravity-based research in this area, as in protein crystal growth.

### PROGRESS DURING FY 1994:

Progress is summarized separately for the three areas listed above.

1. AFM studies of adsorption: We have studied both kinetic and equilibrium aspects of adsorption of positively charged latex particles on mica, which is negatively charged; of particular interest is the effect of ionic strength. Adsorption kinetics are observed to be diffusion-limited. The equilibrium structures display relatively little periodicity, but a fair degree of long-range order; in particular, the interparticle spacing decreases with increasing ionic strength, and hence surface coverage increases with increasing ionic strength. This trend is opposite to that observed for proteins, and the explanation for this could shed considerable light on the mechanisms involved in array formation and, more generally, on protein crystallization. This aspect is still under investigation, but a likely reason lies in the relative contributions of electrostatic and van der Waals interactions to the overall interaction energy.
2. Interaction energy calculations: Our calculations of interaction energy and adsorbed amounts for arrays of spheres have been completed (paper to appear in *Langmuir*). Work to extend these calculations to more realistic arrays of protein molecules is in progress.
3. Surface force measurements: We have successfully performed measurements of the interaction energy between an immobilized array of streptavidin molecules and a lipid bilayer coated on mica. These measurements show evidence of long-range repulsion, presumably due to electrostatic interactions, but a more detailed interpretation and comparison with protein - surface electrostatics computations is difficult because the charge on the bilayer side, which is actually due to the residual charge on the underlying mica, cannot be fixed reproducibly.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 4/94 EXPIRATION: 4/95

PROJECT IDENTIFICATION: 962-23-08-30

NASA CONTRACT NO.: NAG8-1038

RESPONSIBLE CENTER: MSFC

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*Crystallization Studies in Microgravity of an Integral Membrane Protein: The Photosynthetic Reaction Center*

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PRINCIPAL INVESTIGATOR: Dr. James R. Norris

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## CO-INVESTIGATORS:

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TASK OBJECTIVE:

The objective of this project is to determine the effects of microgravity on the crystallization of integral membrane proteins.

## TASK DESCRIPTION:

Advances in the understanding of membrane protein crystallization are important from both biological and pharmaceutical viewpoints. Membrane proteins are responsible for many of the major biological processes such as vision, nerve conduction, cell differentiation, photosynthesis, and respiration. Previous crystallization experiments in microgravity have suggested that the elimination of density-driven convection and sedimentation may generally lead to larger, better-ordered crystals for water-soluble proteins. The potential beneficial effects of microgravity on the crystallization of integral membrane proteins have not yet been tested. The necessity of maintaining membrane proteins in detergent micelles during the crystallization process suggests that the deleterious effects of gravity-driven convection may be more significant during the crystallization of membrane proteins than water soluble proteins.

The effects of microgravity on crystallization of membrane proteins will be tested using the *Rhodospseudomonas viridis* and *Rhodobacter sphaeroides* photosynthetic reaction centers as test proteins. The two reaction centers differ in the proportion of the accessible surface area that is buried in the micelle environment. The comparison of *Rps. viridis* and *Rb. sphaeroides* crystallization will provide a useful indicator of whether the effects of microgravity can be linked to differences in volume fraction of detergent in the detergent-protein complexes. The spacelight and ground control crystallization experiments will be analyzed by three techniques: optical microscopy, x-ray diffraction, and small angle neutron (SANS) and light scattering. The optical microscopy and x-ray diffraction of crystals will determine the effects of microgravity on crystal growth and molecular ordering. SANS and light scattering studies on reaction center solutions at various extents of completion of crystallization will be used to determine the effects of microgravity on the nucleation events that precede and then continue during the crystallization process. The results of reaction center nucleation and crystallization studies in microgravity will be relevant to the crystallization of integral membrane proteins in general.

Since 1988, despite a great deal of worldwide effort, relatively few other membrane proteins have been crystallized in a form suitable for high-resolution structural studies. To date, only the reaction center structures have been solved. Many other membrane proteins have been crystallized, but the generally small crystal size and poor diffraction quality has prevented structural determination. A complicating factor is the presence of the detergent micelle. The effect of the detergents on the crystallization process is unknown. However, the effects of small amphiphiles on crystallization suggest that micelle size or radius may play an important role in determining whether protein-protein contacts predominate to produce a crystalline structure, or whether non-specific micelle-micelle contacts predominate to yield a less ordered array. Crystallization frequently occurs at or near the phase separation of the detergent. This correlation between crystallization and detergent phase separation has led to the investigation of the micelle-micelle attraction, and the role this will play in the early nucleation process. Ultimately, the micelle aggregation must be balanced by protein-protein interactions to result in crystal formation.

# TASK SIGNIFICANCE:

Current results on crystallization in microgravity have shown that the minimization of convection and sedimentation in space leads to improved size and/or ordering of crystals for many water-soluble proteins. In several cases, the resolution of the x-ray diffraction was better than any crystal produced on Earth. In cases where the x-ray diffraction quality did not exceed the best obtained on Earth, the space-grown crystals were still better than those in the control experiment. These results indicate the need for optimization of space hardware compared to the results achieved in the laboratory, but still point to the beneficial effects of microgravity on the crystallization process.

# PROGRESS DURING FY 1994:

1. Determination of Micelle Structure. During this fiscal year this project completed an analysis of detergent micelle structure and micelle-micelle interactions under conditions used for the crystallization of an integral membrane protein, the photosynthetic reaction center. This is the start of a project that will use small angle neutron scattering to determine structures of intermediates in crystallization pathways of integral membrane proteins. SANS was used to analyze the structures of micelles from the detergents octyl- $\beta$ -D-glucoside, OG, and lauryl-dimethylamine-N-oxide, LDAO. To date, only these two detergents have been found to be useful for crystallization of the reaction center. However, crystallization with each detergent requires distinctly different solution conditions. The physical cause for the requirement to adjust the crystallization conditions for each detergent was not previously known, but the differences suggest that detergent properties significantly affect the crystallization process.

Work completed this year (Thiyagarajan and Tiede, 1994 *J. Phys. Chem. in press*) characterized micelle structure and inter-micelle interaction for LDAO and OG in conditions used for protein crystallization using SANS. This work found that LDAO and OG micelles differ significantly in size, sensitivity to heptane triol, and in nature of inter-micelle interactions. Our results suggest that successful crystallization methods can be rationalized in terms of an optimization of micelle size, number density, flexibility of micelle radius of curvature, and suppression of inter-micelle interactions. LDAO and OG micelles were found to differ significantly in size and shape. The LDAO micelle was found to be best fit as an ellipsoid with semi-axes of 30.6 Å and 19.4 Å, while the OG micelle was found to be spherical with a radius of 22.9 Å. The addition of heptane triol to pure LDAO resulted in the formation of smaller, spherical, mixed micelles with radii in the range 17 Å to 21 Å, depending upon conditions. The results suggest that both micelle size and curvature restrictions may contribute to the incompatibility of LDAO for protein crystallization in the absence of additional amphiphiles. The mixed OG-heptane triol micelle was found to be significantly smaller than that with LDAO, having radii in the range 15 Å to 18 Å depending upon conditions, and exhibited a greater number density increase. Evidence was found for interaction between OG and polyethyleneglycol, PEG, that prevents micelle aggregation at high ionic strength, and likely contributes to the particular success of PEG as a protein precipitant when OG is used as the solubilizing detergent. These measurements suggest that the chemical constituents in membrane protein crystallization can be manipulated to optimize micelle size, number density and inter-particle interactions.

2. Protein Aggregation States. During this fiscal year we also initiated SANS investigations that will eventually be used to characterize structure and aggregation state of the reaction center protein as a function of its progress through the crystallization process. During this year, initial focus was on determining the solution structure of the reaction center as a function of crystallization conditions before super-saturated states are reached. SANS data acquisitions were completed on reaction center aggregation states in OG and LDAO, and as a function of ionic strength in these detergents. These measurements found a striking dependence of reaction center aggregation state on detergent and ionic strength. The SANS results suggest that the reaction center exists in an equilibrium between a monodispersed form and an unusual linear aggregate. These studies found that the equilibrium is shifted far towards the linear aggregate with LDAO as the solubilizing detergent at low ionic strength, but shifts to the monodispersed form at high ionic strength (1 M NaCl). Conversely, with OG as the solubilizing detergent the equilibrium was found to be shifted towards the aggregate at high ionic strength, while the monodispersed form is seen to predominate at low ionic strength. While further structural and physical chemical analyses are currently underway, these results show that the existence of the monomeric reaction center state in each detergent is co-incident with



conditions that eliminate micelle interactions. This work is uncovering the connections between micelle properties, protein solubilization and crystallization.

Preliminary SANS data was also acquired on reaction center aggregation in OG\PEG solutions that represent conditions at the start of a crystallization experiment. Although further, ongoing analysis and data acquisition are needed, the preliminary results clearly demonstrate that addition of PEG at concentrations below that needed to induce reaction center crystallization induces a significant aggregation of the reaction center. This data provides evidence that a majority of the reaction center population is likely to exist in aggregates in the dimer to decamer size range at the start of the crystallization process. This data provides evident for the importance of the aggregates states either as competitors to productive crystallization pathways, or possibly as functional intermediates on the actual crystallization paths. Ongoing work will distinguish between these possibilities.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 962-23-08-29

NASA CONTRACT No.: H-13058D

RESPONSIBLE CENTER: MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Thiyagarajan, P., and Tiede, D.M. SANS studies of structure and interactions of detergent micelles as applicable to membrane protein crystallization. Amer. Cryst. Assoc., vol. 22, 75 (1994).

## II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

### *Shear Sensitivities of Human Bone Marrow Cultures*

PRINCIPAL INVESTIGATOR: Dr. Bernhard O. Palsson, Ph.D.

University of Michigan

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective of this research is to reconstruct human bone marrow tissue *ex vivo* using the NASA bioreactor to provide the culture environment for three-dimensional growth.

#### TASK DESCRIPTION:

The specific aims of this program are three:

- 1) To find optimal growth conditions for human bone marrow as a function of the supplied growth factors;
- 2) To develop a shear stress chamber that measures the shear stress sensitivity of human bone marrow cells;
- 3) To use the information gained from 1) and 2) to implement long-term, continuous bone marrow cultures in the RWV.

#### TASK SIGNIFICANCE:

This study will focus on the elucidating role of three-dimensionality in bone marrow stem cell differentiation. The development of a three-dimensional *in vitro* cell model will permit investigation into the biochemical signals that triggers cell differentiation into various stages and subtypes of human blood cells. It also will permit the investigation of the role of three-dimensionality in extensive cell to cell contact and exposure to growth factors.

#### PROGRESS DURING FY 1994:

Conditions for the expansion of adult human bone marrow mononuclear cells in the rotating wall vessel (RWV) have been established. Total cell numbers and the number of progenitor cells increase over time by a factor of 10-20 fold. Shear sensitivity of the cells does not hamper growth of these cells. At present, it is unknown whether stem cells are maintained or if they expand in number in the RWV. This issue will be investigated in the coming year.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 11/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-23-01-09

NASA CONTRACT No.: NAG9-652

RESPONSIBLE CENTER: JSC

*Microgravity Crystallization of Avian Egg White Ovostatin*

PRINCIPAL INVESTIGATOR: Dr. Marc L. Pusey

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

D. Carter

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The research objective is designed to study the ovostatin from the standpoints of its suitability as a model protein for protein crystal nucleation and growth mechanisms, the bio-mechanical movements which apparently form the basis of its inhibitory activity, and using it as a means of studying the  $\alpha_2$ M group of proteins. Further, the intention is to develop the crystallization conditions to be tried based upon studies of the physico-chemical parameters, such as solubility phase behavior and observed nucleation kinetics. This will be our first demonstration protein for the application of what is being learned about the crystal growth of macromolecules.

**TASK DESCRIPTION:**

The study of ovostatin is of interest for several reasons. This protein is attractive as a model for protein crystal nucleation and growth studies. Crystals can be easily and rapidly grown in bulk solution. With a minimum step height of 25-34 nm, surface features should be readily detectable in real-time using interferometric techniques, allowing direct observation of the crystal growth process. The large size would facilitate protein crystal nucleation studies using light scattering methods. Practically, ovostatin can be easily purified in large quantities, requiring about 6 dozen hen egg whites/gram of protein. Material purified has been stable for prolonged periods (over four months) during crystallization trials and has been kept for over one year as a lyophilized powder. Finally, there is the similarity between it and  $\alpha_2$ M. Currently, the only structural information extant is from electron microscopy studies of isolated molecules.

Task 1: Preparation of ovostatin to 1/94 for protein to be used in flight experiment(s). Large scale protein purification will be continuous for the duration of the project. This material will be used to establish the reference ground-based crystal quality against which future flight crystals will be measured. Ovostatin prepared in this laboratory will be made freely available to other researchers wishing to study it.

Task 2: Screening of crystallization condition to 7/93. The best crystallization conditions determined at this time will be used for future flight experiment(s). These conditions will then be used for the final solubility diagram determinations to be used in designing the flight experiments.

Task 3: Establishing baseline crystal quality. Multiple data sets will be acquired using the x-ray diffractometer. These will be used to establish the baseline ground-grown crystal quality.

Task 4: Studies of ovostatin. These will last for the duration of this project. These will be done using the instrumentation developed in the laboratory for the study of the protein crystal nucleation and growth processes.

**TASK SIGNIFICANCE:**

Any knowledge gained about ovostatin based on crystal structure analysis would advance the overall knowledge of this family of proteins.

### PROGRESS DURING FY 1994:

Previous microgravity protein crystal growth experiments have used vapor diffusion, free interface diffusion, and dialysis techniques with most being done using vapor diffusion. Littke and John, using free interface diffusion, reported a  $10^3$  volume increase for beta-Galactosidase over crystals grown on Earth. In vapor diffusion experiments, at least three proteins have shown significantly improved diffraction resolution over any of the crystals grown on Earth, despite the fact that, because of short flight durations, crystallization conditions have had to be optimized for rapid nucleation and growth, i.e., the temporal conditions were more favorable for lower quality crystals. Current experiences are that ovostatin crystals nucleate and finish growing within a one- to three-day period. The process will have to be slowed down to fit better within the timeframe for a typical microgravity PCG experiment, as opposed to the usual case where the crystal growth process must be speeded up. Based on the ovostatin crystal currently grown, there is considerable room for improvement in both crystal size and diffraction resolution.

Ovostatin purified in this laboratory has now been supplied to four other research groups as a test material. Dr. William Wilson (Mississippi State University) has found a dramatic dimerization-tetramerization behavior at low ionic strength. This has led us to an investigation of ovostatin activity vs structure which has been carried out by a mummer faculty (Dr. D. Moriarity, UAH). Ovostatin crystallizability has been found to be very sensitive to the purification procedure and materials employed. We are currently supplying ovostatin to the laboratory of Dr. David Blow (Imperial College) for extensive crystallization trials which we hope will lead to a preliminary structural determination.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-08-26

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

*Study of Crystallization and Solution Properties of Redesigned Protein Surfaces*

PRINCIPAL INVESTIGATOR: Prof. David C. Richardson

Duke University Medical Center

CO-INVESTIGATORS:

M. Hecht

Princeton University

K. Gernert

Duke University Medical Center

J. Richardson

Duke University Medical Center

**TASK OBJECTIVE:**

The objective of this work is to determine what combinations of protein surface features and solution factors best promote the growth of highly ordered protein crystals and to seek strategies for improving control over the process. The experiment design is to make detailed analysis of crystal contacts in Cytochrome B562, construct a planned series of mutations at those crystal contacts study crystal growth and solubility properties of the mutant proteins study resultant crystal order and use the results to further refine analyses, hypothesis, and test mutants.

**TASK DESCRIPTION:**

High-resolution x-ray crystal structures of proteins are the backbone of academic and industrial research efforts to understand and control the detailed functional properties of these important molecules. Many such studies are stymied, either by lack of crystals altogether or by crystals whose degree of order is inadequate to show details at the level of resolution needed.

Two sets of factors jointly influence the growth of ordered protein crystals: the atomic-level details of the protein surface, including flexibility and bound waters; and the solution conditions, including concentration and identity of precipitants and other components, pH and ionic strength, temperature, interactions at solid or liquid interfaces, vibration, convection currents, etc. Traditionally, the protein surface was not variable except by trying different species, and the only strategy for solution conditions was simply trying as many variations as possible. The most general conclusion was that what worked for one protein was likely to be different than what worked for another. Recently, however, there has been support and encouragement, largely led by NASA, for scientific study of the process of crystallization. The most notable single result so far has been the demonstration that growth in microgravity can produce significantly better-ordered crystals for many proteins, presumably because of the absence of convection currents at the crystal surface. The absence of convective currents, in turn, allows for the increased effect of random diffusion of the protein molecules at the crystal surface and for the increased effective binding energy of the protein molecule to the growing crystal. Both of these are presumed to lead to more accurate and more stable attachment of the protein molecules to the crystal, and thus a better-ordered crystal.

Three to four mutants will be studied per year. Each study will involve computer-aided graphics studies of crystal contacts and design of mutants, genetic engineering of mutants, protein purification, crystallization experiments, crystal solubility determinations, face growth rate measurements, calorimetric measurements of crystal growth, and evaluation of crystal diffraction.

**TASK SIGNIFICANCE:**

The research aims are to contribute to the scientific understanding and the practical improvement of protein crystal growth by tying together a series of designed mutations at known crystal contacts with the changes in crystallization behavior and parameters. There are several logical levels at which the results of this research should be useful.

A research study of the relative strengths of the binding of protein molecules into their crystal is important for understanding which factors are improved in microgravity, whether the growth cessation phenomena can be

alleviated in normal gravity, and how changes in crystal contacts can improve the overall order of protein crystals. A detailed study of protein crystal contacts and their specific effects may also help in the future to sort out influences on nucleation of protein crystals versus later growth. This research is designed to collect two overlapping but distinct types of information: what specific side chain changes will strengthen or weaken a contact for a particular crystallization media; and what contact strength is optimum, relative both to the other contacts and to the diffusion and convection conditions. Such information will surely aid in future rational control of the crystallization process.

### PROGRESS DURING FY 1994:

The study of the solubility of wt-B562 crystals (grown under 3.5 M PO<sub>4</sub>, pH 6.8) continues under a variety of buffer conditions, additives and temperatures. A large set of proteins with random mutations to turn C (residues glu-81, gly82, lys+83 wt) were generated, and 39 were sequenced (Hecht lab). The sequenced proteins were characterized by spectroscopy, CD and denaturation studies. From this set, eleven proteins were chosen for additional crystallization studies. First, mutant LGR-B562 has been found to grow diffraction quality crystals from 3.375 M PO<sub>4</sub>, pH 6.8, 1% MPD. Approximately 100 mg of LGR-B562 has been purified to begin solubility measurements. Additionally, approximately 20 mgs of each of the other ten proteins has been purified, and initial crystallization trials of each have been analyzed. One, LAA-B562, has resulted in large diffraction quality crystals, at conditions similar to wt-B562 (3.75 M PO<sub>4</sub>, pH 6.0). Two other proteins (RGM-B562 and SSR-B562) have resulted in small, yet potentially diffraction quality crystals, in conditions neighboring that of wt-B562 (3.75 M PO<sub>4</sub>, pH 6.0, for both). Four other mutants grew small crystals, either plates or rods, in the presence of needles, while the remainder of the mutants formed needles or microcrystallin precipitate.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 8/93 EXPIRATION: 8/96

PROJECT IDENTIFICATION: 962-23-08-22

NASA CONTRACT No.: NAG8-966

RESPONSIBLE CENTER: MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Presentations

Richardson, D.C. "Redesigning protein surfaces and crystal contacts." Poster presented at the Duke University Medical Center Biochemistry Retreat, Beaufort, NC, November 6-8, 1994.

Richardson, D.C. "Study of crystallization and solution properties of redesigned protein surfaces." NASA Protein Crystal Growth Principal Investigator Planning Meeting, Panama City, FL, April 22, 1994.

*Convective Flow Effects on Protein Crystal Growth and Diffraction Resolution*

PRINCIPAL INVESTIGATOR: Prof. Franz E. Rosenberger

University of Alabama, Huntsville

CO-INVESTIGATORS:

L. Monaco

University of Alabama, Huntsville (UAH)

**TASK OBJECTIVE:**

This research aims at developing a detailed understanding of

- the nucleation and growth mechanisms involved in the crystallization of globular proteins,
- the formation mechanisms of structural and compositional nonuniformities (defects) in protein crystals and their dependence on growth conditions, and
- the dependence of x-ray diffraction resolution of protein crystals on defect types and concentrations and, thus, on crystallization conditions.

**TASK DESCRIPTION:**

We seek to establish, for select proteins, a correlation of well-defined solution conditions (purity, pH, buffer, precipitant, supersaturation, temperature, and bulk transport) with nucleation and growth behavior during crystallization, and the x-ray diffraction resolution of the resulting crystals.

Towards the above goals we are pursuing the following tasks:

- Characterization of the purity of protein solutions by gel electrophoresis. Preparation of highly homogenous protein samples by high pressure liquid column chromatography.
- Studies of protein interactions and aggregation in under- and supersaturated solutions by static and dynamic light scattering.
- Determinations of the precipitant repartitioning between solutions and growing protein crystals, using atomic absorption and optical spectroscopy, and ion selective potentiometry.
- In-situ studies of the protein growth morphology and kinetics, with and without forced solution flow, by high-resolution microscopic interferometry.
- Numerical modeling of diffusive-convective mass and momentum transport in geometries characteristic of protein crystallization on Earth and in space, using experimentally determined precipitant repartitioning and growth kinetics data.
- Measurements of protein/precipitant diffusivities in saturated and supersaturated solutions, employing a novel interferometric technique that requires only small solution volumes.
- Measurements of the kinematic viscosity of supersaturated protein solutions, with a capillary flow technique.
- Determination of protein solubilities as a function of precipitant concentration, pH and temperature, using a miniaturized optical scintillation technique.
- Characterization of the structural quality of selected crystals by x-ray diffraction and topography to reveal the influence of kinetics and transport effects.

**TASK SIGNIFICANCE:**

The pharmaceutical industry needs protein structure information to facilitate rational drug design. However, many of the currently available protein crystals are too imperfect to yield detailed structure information. The reasons for this low crystal perfection are not well understood. Interestingly, crystallization experiments in space have led to significant improvements in crystal perfection for some proteins. Again, the physical mechanisms for this are not clear. Our research aims at clarifying the connection between the magnitude of gravity present during protein crystallization and the resulting crystal quality. This insight is expected to lead to the design of protein crystal growth techniques that result in larger and more perfect crystals.

## PROGRESS DURING FY1994:

Our electrophoretic measurements revealed that significant amounts of high molecular weight protein impurities exist in all commercial lysozyme stocks. These stocks are widely used without further purification in protein crystallization studies. The removal of these impurities, which appear to influence most kinetics and repartitioning processes, is being pursued with fast protein liquid chromatography.

Simultaneous static and dynamic light scattering measurements were performed on buffered solutions with lysozyme solutions of various protein and NaCl concentrations. Contrary to most earlier work, the lysozyme diffusivities were found to either increase or decrease with lysozyme concentrations, depending on the salt concentrations. The corresponding static light scattering intensities show opposite dependence on lysozyme concentration. These data are compatible with concentration-dependent changes of monomeric interactions. At the same time, these findings appear inconsistent with earlier claims of the formation of small protein aggregates prior to nucleation.

The high-resolution microscopic investigations of lysozyme growth morphology and kinetics without forced flow have revealed several novel phenomena. Depending on the impurity content of the solution, the growth step density is either higher or lower at the periphery of a crystal facet than in its center. We also found growth kinetics oscillations even at stable growth temperatures. These oscillations, which increase in amplitude with supersaturation and crystal size, are associated with the formation of compositional inhomogeneities. These phenomena form the first indication for mechanisms by which bulk transport conditions and, thus, gravity could influence the protein crystal perfection.

The repartitioning of  $\text{Na}^+$  and  $\text{Cl}^-$  ions between lysozyme solutions and crystals was investigated for a wide range of crystallization conditions. A nucleation-growth-segregation model was developed to interpret the large body of data in a unified way. The results strongly suggest that lysozyme crystals possess a salt-rich core with a diameter on the order of 10  $\mu\text{m}$ . These results are corroborated by white beam x-ray topographs (obtained on our crystals by V. Stojanoff at the Brookhaven National Laboratories) which show an accumulation of defects in the crystals' historical center. Further support for coring comes from our preliminary investigations of compositional inhomogeneities on cross-linked, microtomed crystals with scanning electron microscopy and energy dispersive spectroscopy. In addition it appears that the high molecular weight impurities preferentially partake in the coring. Although these findings have to undergo further scrutiny, they suggest some far-reaching connections between protein crystal perfection and crystal growth conditions.

A realistic numerical model for diffusive-convective transport in lysozyme crystallization has been completed. The results indicate that even in the small crystallization cells typically employed, protein concentration nonuniformities and gravity-driven solutal convection can be significant. The calculated convection velocities are of the same order of magnitude as those found in earlier experiments. As expected, convective transport enhances the growth rates. However, even when diffusion dominates mass transfer, i.e. at zero gravity, lysozyme crystal growth remains kinetically limited. The salt distribution in the crystal is predicted to be nonuniform at both 1 g and 0 g, as a consequence of protein depletion in the solution.

To provide realistic input for the modeling, and to link light scattering results with macroscopic diffusivity data, an interferometric diffusivity measurement setup has been completed. This setup requires only small (protein) solution volumes and is accurate to better than 1%, as shown by extensive tests with NaCl solutions.

Also in support of the numerical modeling, the viscosity of a large number of lysozyme solutions was monitored over time with a temperature controlled Cannon-Fenske viscometer under conditions which lead to crystallization. Changes in the kinematic viscosity of aqueous solutions by 1% could be detected. However, none of the protein solutions studied showed a change in kinematic viscosity as the solutions aged before crystallization. This indicates the absence of a significant concentration of large clusters in lysozyme solutions prior to nucleation.



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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 2/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-08-23

NASA CONTRACT No.: NAG8-950

RESPONSIBLE CENTER: MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Fredericks, W.J., Hammonds, M.C., Howard, S.B., and Rosenberger, F. Density, thermal expansivity, viscosity and refractive index of lysozyme solutions at crystal growth concentrations. *J. Cryst. Growth*, vol. 141, 183-192 (1994).

Rosenberger, F. Boundary layers in crystal growth. *Prog. Crystal Growth Charact.*, vol. 26, 87-98 (1993).

Rosenberger, F. Short duration low-gravity experiments - time scales, challenges and results. *Micrograv. Sci. Technol.*, vol. VI, 142-148 (1993).

**Presentations**

Rosenberger, F. "Interplay between anisotropic interface kinetics and nutrient bulk transport." Plenary lecture at the Annual Conference of the German Mineralogical Society, Freiberg, Germany, September 17-23, 1994.

*Enhancement of Cell Function in Culture by Controlled Aggregation*

PRINCIPAL INVESTIGATOR: Prof. W. M. Saltzman

Johns Hopkins University

CO-INVESTIGATORS:

Prof. K. Leong

Johns Hopkins University

**TASK OBJECTIVE:**

Cell-cell contact within tissues is fundamental to the regulation of cell differentiation and function. Cell aggregates, formed *in vitro* and maintained in culture, have been shown to maintain many of the functions of the native tissue. The main objectives of this program are: i) development of methods for controlling cell aggregation using bioactive polymers and polymer microspheres and ii) systematic study of the function and behavior of suitably aggregated cells in culture. The discovery of new methods for improving cell growth and function in culture is critical to the development of hybrid artificial internal organs and mammalian cell bioreactors.

**TASK DESCRIPTION:**

To achieve these goals, we have formulated the following specific objectives for a three-year period of study:

**Objective 1: Synthesis of water soluble polymers for controlling cell aggregation.** We will synthesize water soluble polymers with bioactive groups that are specifically recognized by certain cells and use these multifunctional polymers as molecular nuclei to initiate and control cell aggregation. We will use these polymers to control aggregation by adding them to gently agitated or quiescent suspensions of single cells; this technique will produce small cellular aggregates (<10 cells, diameter <100  $\mu$ m). By altering the properties of the polymers and the conditions of aggregation, we will identify approaches for obtaining cell aggregates of different size, polydispersity, and morphology. We have demonstrated the feasibility of this approach using N-acetyl glucosamine (specific for chicken hepatocytes) attached to vinyl polymers.

**Objective 2: Fabrication of polymer microspheres for carrier-assisted cell aggregation.** We will fabricate microspheres containing encapsulated, soluble mediators of cell growth and function. We will use the microspheres to create larger aggregates with a central polymer core. We have also demonstrated the feasibility of this approach using microspheres composed of vinyl polymers and cultured hepatocytes.

**Objective 3: Development of methods for culturing cell aggregates under unit gravity.** In preliminary studies, we have encapsulated hepatocytes and hepatocyte aggregates within gels of type I collagen, cultured these encapsulated cells, and examined subsequent cell growth, function, and viability. We will test gels of collagen under different hydration conditions to find an optimal experimental system for maintaining cells in an aggregated and suspended state in the laboratory. The gels will be used to suspend aggregates created with water-soluble polymers and microspheres. To develop model culture systems representing both liver and neuronal tissues, we will use primary cultures of hepatocytes, primary cultures and fetal brain cells, neuroblastoma cell lines, and the PC12 cell line.

**Objective 4: Measurement of cell function under different aggregation conditions in gel culture.** We have already developed methods of monitoring cell growth and metabolism in culture by measuring cellular protein, DNA, and lactate dehydrogenase (LDH) content. We have also developed methods for monitoring cell function in culture by following albumin and uric acid secretion (for hepatocytes) and expression of specific enzymes and responsiveness to nerve growth factor (for PC12 cells). Using the optimal experimental system, defined in specific objective 3, we will systematically examine the function of cell aggregates in culture. Aggregates will be cultured under a variety of conditions including i) conventional static culture, ii) static culture with aggregates suspended within gels of extracellular matrix molecules, and iii) microgravity culture of aggregates within the NASA rotating-wall bioreactors.

**Objective 5: Preparation of experiments for evaluation under microgravity conditions.** In specific objectives 1 through 4, we will have identified the important variables for controlling cell aggregation and function in aggregate culture. In the final stages of this project, we will design methods for developing physiologically realistic cell aggregates under microgravity conditions and for testing the influence of aggregation on cell function in suspension culture under microgravity in space.

**TASK SIGNIFICANCE:**

These studies are uniquely suited for study in microgravity. First, cell aggregation in zero gravity will be driven by migration and diffusion rather than by forced collisions as is necessary on earth. The resulting aggregates may be closer to those found in tissues, since tissues are formed by migration and selective adhesion. Second, gentle suspension culture techniques can be used to culture the aggregates in microgravity. By the end of the period of laboratory study proposed here, we will have developed cell aggregation techniques appropriate for testing under microgravity conditions in space.

**PROGRESS DURING FY 1994:**

Objective 1: We have synthesized several water-soluble molecules as molecular nuclei for cell adhesion. These molecules are based on poly(ethylene glycol) with bioactive peptides,  $\text{NH}_3$ -Gly-Arg-Gly-Asp (GRGD) and  $\text{NH}_3$ -Gly-Tyr-Ile-Gly-Ser-Arg (GYIGSR), grafted to the termini. We have developed covalent coupling methods for attaching these peptides to PEG with a variety of molecular weights (5,000 to 30,000) and to polystyrene microspheres (0.1 to 10  $\mu\text{m}$ ). We have used quantitative cell adhesion assays to determine the best peptides for conjugation. We have examined the kinetics of cell aggregation of a number of important cell types (including neural cells and cell lines, fibroblasts, and genetically engineered fibroblasts). We are developing methods for examining cell function for aggregates maintained in culture. The first report in this work was published this year (Dai, Belt, and Saltzman, 1994) and other publications are in progress.

Objective 2: We have synthesized polystyrene polymers modified with carbohydrates and peptides at the surface. A manuscript describing this work was published this year (Gutsche, et. al., 1994). These unique polystyrene-based polymers can be formed as microspheres, and cells attach and grow avidly to these novel microcarriers. We have developed similar methods for coupling peptides to polystyrene supports with a range of sizes.

Objectives 3 and 4: We have developed methods for culturing and forming aggregates under unit gravity. A manuscript that was recently accepted for publication is appended. (Krewson, C.E., Chung S.W., Dai, W., and Saltzman, W.M. *Biotechnology and Bioengineering* 43:555-562 (1994). We have adapted these methods for producing aggregates within the NASA rotating-wall vessels.

Objective 5: Only applicable to the third project year.

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**STUDENTS FUNDED UNDER RESEARCH:**

TASK INITIATION: 11/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-23-01-10

NASA CONTRACT NO.: NAG-654

RESPONSIBLE CENTER: JSC

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Saltzman, W.M., Belt J., Dai W., Krewson C.E., Livingston T. Parkhurst M.R. and Shin M. "Synthesis of polymeric substrates and characterization of cell-polymer interactions." Keystone Symposium on Tissue Engineering, Taos, NM. February 1994.

Saltzman, W.M., Dai W. Krewson C.E. and Parkhurst M.R. "Polymers for controlling cell motility and aggregation." 1st International Conference on Cellular Engineering, Stoke on Trent, UK, 12-15 September 1993.

*Culture of Porcine Islet Tissue: Evaluation of Microgravity Conditions*

PRINCIPAL INVESTIGATOR: Dr. David W. Scharp

Washington University School of Medicine

Co-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Porcine islet tissue is receiving new attention as an attractive, potential tissue for application in clinical islet transplantation in patients with Type 1, Insulin-Dependent Diabetes Mellitus (IDDM). Recent clinical success of islet transplantation into patients with renal grafts using human islets and standard immunosuppression demonstrated that greater than one year islet graft function can be achieved off insulin therapy. As additional patients are being transplanted to establish how many patients can achieve insulin independence and for what duration, investigators are anticipating that the 4,000 human organ donors a year will be insufficient for the numbers of potential transplants that can be achieved. Thus, adult porcine islet isolations are being developed. We have developed an adult porcine islet procedure but realize that the use of neonatal porcine islet tissue is more suited for clinical application from a cost production viewpoint as well as from a safety viewpoint, since the neonatal porcine islet tissue could be obtained from gnotobiotic donors. While this seems ideal, there has not been any reliable way to isolate neonatal islet tissue in any quantity nor any method to culture these islet cells. We have recently developed a markedly improved method for the isolation and purification of neonatal porcine islet tissue, but, have no reliable method to culture islet cells that can not only produce new islets, called pseudoislets, but also take advantage of their inherent growth and maturation potential prior to consideration of transplantation.

Learning of the microgravitational culture system with low shear rate and the proven importance in producing other tissue types from single cells developed by Dr. Glenn Spaulding (NASA JSC), he and I have established a new collaboration that would combine our islet tissue.

**TASK DESCRIPTION:**

To accomplish these three objectives, we propose the following specific aims for the investigations:

1. To culture neonatal islet tissue by rotational, microgravitational and static methods to determine optimal ways of formation and preservation of functional pseudoislets.
2. To examine the ability of cultured neonatal porcine islet tissue to develop and maintain differentiated islet functional characteristics.
3. To determine the ability of optimally cultured porcine islet tissue to be successfully transplanted into diabetic recipients.
4. To examine the replication potential of neonatal porcine islet tissue through culture manipulations. These proposed studies combine the islet expertise with a method of rotational islet culture that has successfully formed neonatal porcine pseudoislets with the microgravitational expertise with a specific low shear culture system that seems ideally suited to the fragile neonatal islet cells. Successful completion of these proposed studies should provide important results that will have considerable application in the islet field as well as in the field of microgravitational studies important to future NASA objectives.

**TASK SIGNIFICANCE:**

The results of these studies will provide new information to three areas. The results will be important to: 1) a better understanding of the development of neonatal islet tissue, 2) NASA considerations of islet tissue as a potential type of tissue for their microgravitational studies, and 3) islet transplantation for developing an effective culture system for this promising new source of islet tissue.

### PROGRESS DURING FY 1994:

This project continues to utilize islet tissue isolated from 14 to 29 day neonatal pigs which have been surgically altered at three days of age and given feed enriched with potential growth factors to increase the beta cell production and maturation for the 10-day to two week period prior to harvest. So far this year, we have performed twelve separate experiments, each closely monitored at different stages by sampling for DNA extraction, insulin production, glucose utilization rates, insulin extraction, and histology evaluation.

Results show a two- to seven-fold increase in the number of aggregates at two weeks of culture. Although glucose utilization is fairly constant, there seems to be a maximum output of insulin at one week of culture. Final viability testing shows functional islet tissue and a 95% viability by fluorescein diacetate staining. Recent studies have been focused on varying the culture medium used, as well as varying the tissue and bead ratios. One of our latest developments has to do with increasing the ability to process more tissue at a time, therefore cutting down on the variabilities and increasing the yield per grams of tissue, allowing us to culture more aggregates from each experiment.

Although *in vitro* testing has been a successful tool in monitoring the progress of our tissue survival and growth, we have just recently begun to study the potential of this project with *in vivo* growth of our neonatal tissue. We are using the SCID (sever-combined immune deficient) mouse as a recipient model, in order to avoid any rejection problems. The results of these experiments are too recent to draw conclusions but our expectations for this series are high.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-01-08

NASA CONTRACT NO.: NAG-653

RESPONSIBLE CENTER: JSC

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*Automation of Protein Crystallization Experiments: Crystallization by Dynamic Control of Temperature*

PRINCIPAL INVESTIGATOR: Dr. Keith B. Ward

Naval Research Laboratory (NRL)

CO-INVESTIGATORS:

W.M. Zuk

Geocenters, Inc./Naval Research Laboratory (NRL)

M.A. Perozzo

Naval Research Laboratory (NRL)

**TASK OBJECTIVE:**

The goal of this research program is to develop a dynamically-controlled crystallization system (DCCS) in which protein supersaturation is controlled by varying the temperature while crystallization is monitored by optical means. This device will also be capable of being controlled telerobotically. The program intends to extend its accomplishments in this area by continuing to enhance the DCCS, expanding the system to include multiple crystallization chambers and incorporating more efficient and versatile systems for monitoring the progress of nucleation and crystallization. A final goal of this project is to ascertain to what extent the technique of temperature-controlled crystallization is applicable for protein crystallization.

**TASK DESCRIPTION:**

A study of a representative sample of well-characterized proteins that have been successfully crystallized using other methods is proposed. The temperature coefficient of solubility will be measured using the DCCS, and attempts to prepare crystals in this apparatus will allow us to judge the general usefulness of this approach.

The proposed methods of research include further modifying the current design to incorporate video monitoring to provide visual observation of growth volumes, to introduce dynamic light scattering, and to expand the system to include multiple crystallization cells, each with separate temperature controls. Telerobotic control experiments will continue using enhanced control software, and the results of the experiments will be aimed at defining the capabilities and limitations of remotely-controlled crystallization protocols on space platforms in microgravity. Collection of protein temperature solubility data will be enhanced by the development of more fully automated software algorithms. The temperature of a sample is slowly changed step-wise until the level of scintillation signal indicates that the crystallization phase boundary has been crossed. The temperature will then be recycled using a finer step size until the solubility temperature of the sample is determined to within 0.1 °C. Although currently some of this process is conducted manually, further development of the control software will completely automate the process.

**TASK SIGNIFICANCE:**

This research is important in continuing the development of dynamically-controlled crystallization systems, proving the usefulness of dynamic control in conducting protein crystallization experiments in microgravity. This work will also aid the current effort of other NASA-funded Principle Investigators in designing advanced crystallization apparatuses. This system will also be used, while it is being developed, to explore whether temperature control of supersaturation is a technique that can have wide applicability in laboratory-based protein crystallization.

**PROGRESS DURING FY 1994:**

The DCCS has been miniaturized and redesigned to allow the use of crystallization solutions as small as 100 microliters. Temperature is controlled through the use of small thermoelectric coolers. The new device utilizes a common miniature fluorimeter cell as the growth chamber, greatly easing the preparation and clean-up of experiments. The system was also designed to permit easy integration of a miniature video camera for visual observation of growth cells. Preliminary solubility and crystallization experiments have been conducted to demonstrate the usefulness of the new apparatus. Control software is continually being refined and will soon allow multitasking of procedures so that multiple experiments may be conducted simultaneously.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-23-08-24

NASA CONTRACT NO.: H-07975D

RESPONSIBLE CENTER: MSFC

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*Thermal Optimization of Growth and Quality of Protein Crystals*

PRINCIPAL INVESTIGATOR: Dr. John M. Wiencek

University of Iowa

CO-INVESTIGATORS:

E. Arnold

Rutgers University

**TASK OBJECTIVE:**

The overall goal of this project is to control supersaturation at constant values during protein crystal growth by varying the temperature in a predetermined (by simple theory) manner. Applying the theory requires knowledge about specific physicochemical properties of the protein solution including the effect of supersaturation on growth rates and the effect of temperature on protein solubility. Our specific goals for FY94 were:

- Application of a temperature control strategy which maintains constant supersaturation to the growth of lysozyme crystals and comparison to traditional isothermal strategies.
- Investigation of batch isothermal calorimetry as a tool to determine lysozyme solubility as a function of temperature by employing the Van't Hoff equation.
- Construction of a video microscopy apparatus for determination of crystal growth rates and terminal size.

Work for the coming year will focus on the measurement of solubility and crystal growth rate for human serum albumin as well as the assessment of the crystals grown by the developed strategies via xray diffraction.

**TASK DESCRIPTION:**

Three protein systems have been chosen for study: lysozyme, human serum albumin (HSA), and HIV reverse transcriptase (RT). Each of these proteins have unique features that make them interesting. Lysozyme and HSA represent fairly inexpensive and readily available proteins and will be model systems for investigations elucidating the effects of growth rates on crystal quality. Once strategies that are optimal are available, such strategies will be applied to the RT system as a realistic test case.

Experimental evidence suggests that larger and higher quality crystals can be attained in the microgravity environment of space. Fundamental studies have attempted to measure and model the effects of gravity-induced convection and sedimentation on the crystal growth process. However, the effect of growth rate on protein crystal quality is not well documented. If the growth rate is controlled, how much time is required to allow for interfacial attachment of the large protein molecule? What is the impact of this "attachment time" on crystal quality? This research effort is directed at measuring the effects of crystal growth rate on the ability of crystals to diffract xrays. We hope to link crystal quality to slow growth rates and discern "how slow is slow enough." In addition, processing strategies will be developed which xray crystallographers can use to grow larger, high-quality crystals. The investigation requires the measurement of protein (lysozyme, HSA and RT) solubility at two or more different temperatures (typically 4 ° and 25 ° C) and the growth rate at two or more different supersaturations. Microcalorimetry is a potentially powerful technique to measure these and other (e.g nucleation) physical parameters of the protein systems.

**TASK SIGNIFICANCE:**

Development of a systematic method of protein crystallization may lead to crystallization of previously uncrystallizable proteins and add to current knowledge of protein structure/function relationships. Knowledge of detailed protein structure is essential for rational design of therapies and small molecule pharmaceuticals.

PROGRESS DURING FY 1994:

Material balances and kinetic rate laws were combined with solubility data to produce a temperature control algorithm designed to maximize crystal size and maintain the crystal growth conditions in or close to the metastable zone. Several assumptions were made in the construction of the temperature algorithm, the first being that of constant growth rate. Because our experiments were performed in the reservoir of a temperature-controlled water bath, it was not feasible to measure the crystal growth rate during the course of the experiments. Studies are currently underway to measure the growth rate of crystals *in situ*, to determine whether the assumption of constant growth rate was met in our experimental system. The second major assumption in the temperature control model is that only one crystal is present and growing throughout the experiment. With the exception of the isothermal 4 °C program, the predicted final size of the seeded samples corresponds closely with the measured average final size in the seeded systems, indicating that this may not be a limiting assumption. Nucleation can also be accounted for in future studies by taking additional crystals into account in the mass balance. A third assumption is that the growth rate model is dependent only on supersaturation. This model was used in the generation of four temperature control strategies for the growth of lysozyme crystals: curved (constant growth model), linear ramp, isothermal 20 °C, and isothermal 4 °C. Both the linear and the curved temperature programs yielded large, well-formed crystals. The isothermal 4 °C program also resulted in large crystals, but they were poorly formed due to the high initial growth rates. The enthalpy of crystallization of hen egg-white lysozyme in two 0.05 M acetate buffers (5% NaCl, pH 4.6 and 3% NaCl, pH 5.2) was determined at 15 °C using isothermal calorimetry and was found to be  $-17.1 \pm 3.2$  kcal/mol (5% NaCl) and  $-10.5 \pm 2.3$  kcal/mol (3% NaCl). These values were found to agree within experimental error with the enthalpy of crystallization determined from a Van't Hoff plot of solubility.

In this work, we have shown that the use of controlled temperature changes leads to improved size of lysozyme crystals, and can limit nucleation in a crystallizing system. These temperature ramps must have a basis in the physicochemical properties of the system (i.e., solubility, enthalpy and growth rate kinetics). Perhaps the major limitation in extending the use of this algorithm to other systems is the limited amount of physicochemical data available for most proteins. However, our results show that there is some flexibility in the application of temperature control, so that a linear temperature ramp may be just as effective as our "constant growth rate" algorithm. This indicates that moderately accurate physical and chemical data may be required to generate an appropriate temperature ramp. The use of calorimetric methods to attain solubility and enthalpic data may provide another source of the necessary information. It is expected that as more information becomes available, this type of predictive temperature control strategy can be applied to a wide variety of systems, facilitating the growth of large, single protein crystals suitable for X-ray crystallography.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 3

TASK INITIATION: 6/93 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-23-08-28

NASA CONTRACT No.: NAG8-975

RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Wienczek, J.M. "Thermal optimization of protein crystal growth." NASA Protein Crystal Growth Conference, Panama City Beach, FL, April 26, 1994.

Wienczek, J.M., Schall, C.A., Li, E., and Arnold, E. "Measurement of enthalpy of fusion of lysozyme crystals by calorimetry and application to maximization of crystal size." AIChE Annual Meeting, St. Louis, MO, November, 1993.

*Search for Dilute Solution Property to Predict Protein Crystallization*

PRINCIPAL INVESTIGATOR: Dr. W. W. Wilson

Mississippi State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The primary objective of the research is to discover a unique dilute solution parameter that universally and unambiguously predicts protein crystallization.

Since most crystallographers will not have access to sophisticated laser scattering instrumentation, a secondary objective of this research is to construct a simple laser scattering device that determines the universal predictor values. The device will be miniaturized to work with sub-milliliter volumes and incorporate the latest optical fiber technology for beam delivery and signal detection.

**TASK DESCRIPTION:**

Static, dynamic and electrophoretic laser scattering techniques will be used to carefully measure an array of thermodynamic and hydrodynamic (not kinetic) solution parameters for each of a group of selected proteins dissolved under crystallizing as well as non-crystallizing solvent conditions. The proteins chosen will have a wide variation with respect to molecular weight and crystallizing conditions such as temperature, pH and crystallizing agent type (inorganic salts, PEGs and other organics). The laser scattering solution parameters will be measured in the dilute protein concentration regime, often 10-20 times below protein saturation.

The research approach is to obtain comprehensive measurements of the SLS, DLS and ELS parameters from a set of selected proteins under both crystallizing and precipitating conditions with particular attention given to the dilute solution regime. The selection of the proteins is significant, and some collaboration with protein crystallographers will be required to totally define the set. Prior verbal agreement for such advisory collaboration has been obtained from Marc Pusey and Dan Carter at Marshall Space Flight Center in Huntsville, Alabama, Pat Weber at Dupont in Wilmington, Delaware, Alex McPherson at the University of California, Riverside, Franz Rosenberger at the University of Alabama, Huntsville, and Charlie Bugg and Larry DeLucas at the University of Alabama, Birmingham. Use will also be made of the Biological Macromolecule Crystallization Database compiled by Gary Gilliland at the Center for Advanced Research in Biotechnology in Rockville, Maryland. Based on years of experience in performing laser scattering measurements and on the man-power requested in the budget, a target number of twenty proteins is projected for the set, corresponding to roughly one complete set of measurements per protein per month. This amount of time accounts for protein purification procedures as well as repetitions for each of the SLS, DLS, and ELS experiments.

**TASK SIGNIFICANCE:**

It is anticipated that a particular solution parameter (or combination of parameters) will be discovered that has quantitative values within a reasonable narrow range for crystallizing conditions and values significantly outside that range for non-crystallizing or precipitating conditions. If such a universal predictor can be proven, then its use will have an immediate impact in the protein crystal growth community in general and microgravity research in particular. The solution conditions for protein crystallization in a microgravity environment should be maximized during ground testing so that a high probability for crystallization is achieved. Having a universal predictor will allow crystallographers to fine tune existing crystallization protocol or discover new conditions to crystallize difficult proteins.

**PROGRESS DURING FY 1994:**

The focus of work during this period was investigating three laser scattering methods as potential diagnostics for predicting solution conditions favorable for protein crystallization. One method, electrophoretic laser scattering or ELS, has been found to be generally not appropriate for this work. We have determined that the resolution of ELS for small proteins with molecular weights less than about 100,000 daltons is poor. ELS may be useful for studying large particles such as viruses, but its applicability for protein crystallography is doubtful. Accordingly, our emphasis shifted to static laser scattering (SLS) and dynamic laser scattering (DLS), both of which seem promising. We have studied about fifteen different protein/solvent crystallization conditions by SLS and have found that a thermodynamic solution parameter called the second virial coefficient, B22, seems to be a general predictor for protein crystallization. DLS was used for some of the same protein/solvent pairs to determine if the diffusion virial coefficient, KD, could also be used as a predictor. The results for DLS are not conclusive at this time.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 7/93 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-23-08-21

NASA CONTRACT NO.: NAG8-965

RESPONSIBLE CENTER: MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Journals**

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*Characterization of Solvation Potentials Between Small Particles*

PRINCIPAL INVESTIGATOR: Dr. Charles F. Zukoski

University of Illinois, Urbana-Champaign

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

A combined experimental and modelling effort is used to characterize how variations in solvent chemical potential alter the states of aggregation of colloidal particles.

**TASK DESCRIPTION:**

The objective of the FY94 program is to develop a combined density fractional theory and Monte Carlo simulation technique to describe the phase behavior of particles where interactions are dominated by solvation forces. In addition, the role of solvent/particle interactions in creating hydrophobic attractions is explored. On the experimental side, direct links are made between interparticle forces and phase behavior through measurements of protein second virial coefficients by light scattering and phase behavior as protein concentration is increased.

**TASK SIGNIFICANCE:**

Developing methods which reliably result in high quality protein crystals is of major technological significance in the development of fundamental understanding of biochemical phenomena and the expression of genetically altered therapeutic proteins. In the work carried out here, new methods of controlling protein crystallization are explored. The modelling effort seeks to guide the experimental program by providing understanding of the role of solvation interactions in controlling the state of protein aggregation. The modelling effort has shown that the rarely recognized variable of solvent chemical potential can be used to control protein crystallization behavior. The experimental program is aimed at developing methods of characterizing protein interactions as solvent chemical potential is altered and demonstrating links with crystallization behavior.

**PROGRESS DURING FY 1994:**

Substantial modelling progress has been made. Simulation studies show that variations in solvent chemical potential can strongly influence how small particles associate and the strong influence of solvent/particle interactions. Experimentally, light scattering techniques have been used to determine the second virial coefficient,  $B_2$ , of lysozyme molecules as a function of pH and ionic strength.  $B_2$  is a measure of the strength of interparticle forces trending from positive to negative as the interactions pass from repulsive to attractive. Linking  $B_2$  to conditions resulting in crystallization remains a central task in FY95.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 6/93 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-23-08-27

NASA CONTRACT No.: NAG8-976

RESPONSIBLE CENTER: MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

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**Presentations**

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*Effects of Energy Release on Near Field Flow Structure of Gas Jets*

PRINCIPAL INVESTIGATOR: Prof. Ajay K. Agrawal

University of Oklahoma

CO-INVESTIGATORS:

S.R. Gollahalli

University of Oklahoma

**TASK OBJECTIVE:**

The primary objective of this research is to understand how buoyancy affects the structure of the shear layer, the development of fluid dynamic instabilities, and formation and characteristics of the coherent structures in the near-nozzle regions of burning gas jets. The secondary objective is to understand the role of buoyancy in the flame lifting and reattachment process, evaluate the scaling behavior of diffusion flames, and aid in the development of a theoretical model by providing quantitative temperature data throughout the flame in the absence of buoyancy effects.

**TASK DESCRIPTION:**

The initial phase of this project involves the visualization of near-field flow structures in cold jets and nonsooting flames at the same flow conditions or the same jet exit Reynolds number, allowing the effects of energy release on these structures to be identified by comparison. The experiments will use hydrogen and hydrogen-inert gas mixtures as the fuel with air as the oxidizer. Energy released in the flame will be controlled by varying the hydrogen mole fraction in the jet stream. Attached flames, lifted flames, and flames in the transition region between these two extremes will be studied. Experiments for all cases will be conducted in both normal and reduced gravity.

**TASK SIGNIFICANCE:**

The phenomena occurring near the exit of a gas jet nozzle determine burning characteristics and the rate of pollutant generation. The effect of buoyancy on these processes is poorly understood and hence it is difficult to model when designing commercial combustors. This project will study the fluid dynamics of turbulent gas jet combustion, applicable to commercial combustors of this type and may lead to higher efficiencies and lower pollution generation levels.

**PROGRESS DURING FY 1994:**

Since the award of this grant in June 1994, laboratory space was acquired and safety procedures were established to begin this project.

Computer software for the generation of rainbow filters was developed in conjunction with Dr. Fletcher Miller, Case Western Reserve University, and NASA LeRC equipment and personnel. The filters were printed on a high resolution Polaroid digital film recorder, which is capable of producing 35 mm slides. These slides are being used with a schlieren system interfaced with a 3-CCD color camera and a personal computer equipped with image processing software.

Concurrently, experiments were conducted using hydrogen as the fuel at atmospheric pressure to determine an appropriate size of the nozzle for drop tower experiments. The accompanying fuel supply system and test section for both atmospheric and low pressure studies were designed and fabrication has been initiated.



## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 2

MS Students: 0

PhD Students: 0

TASK INITIATION: 6/94 EXPIRATION: 5/97

PROJECT IDENTIFICATION: 962-22-05-57

RESPONSIBLE CENTER: LeRC

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*Radiative Extinction of Diffusion Flames*

PRINCIPAL INVESTIGATOR: Prof. Arvind Atreya

University of Michigan

CO-INVESTIGATORS:

Prof. I.S. Wichman

Michigan State University

**TASK OBJECTIVE:**

The objective of this program is to quantify the conditions under which a stabilized, laminar diffusion flame will be extinguished by radiative heat losses from flame-generated particulates (e.g., soot) that drain the chemically released energy from the flame. These tests must be conducted in microgravity because radiation-induced extinction may not be possible under normal-gravity conditions where buoyancy-generated convection would sweep the radiating sources upward and away from the flame.

**TASK DESCRIPTION:**

The program is to have simultaneous experimental and theoretical efforts. Experimentally, normal-gravity tests using a quasi-one-dimensional counterflow diffusion flame burner will be studied to quantify soot production and oxidation rates and their optical properties. These data are needed both for the formulation of the reduced-gravity testing and for the development of theoretical models.

Subsequent reduced-gravity testing is to be pursued in the 2.2 second drop tower at NASA Lewis Research Center, where a laminar diffusion flame is to be stabilized about a spherical porous burner. In these tests the local fuel concentrations will be varied by the introduction of inerts into the fuel-flow stream. Measurements of flame temperature and radiation flux will be used for comparisons with theory.

A numerical model will be developed to simulate the reduced-gravity experimental configuration, and will include a chemical-kinetics-model and an empirical model of the production and consumption of soot particulates. A model of the radiant emissions from the flame, associated with the particulates, will be developed.

**TASK SIGNIFICANCE:**

The microgravity flames will demonstrate the concept of radiative extinction in stabilized flames, distinct from the case of spreading diffusion flames. This mechanism of extinction is likely unique in microgravity, and thus may have application to fire safety aboard orbiting spacecraft.

**PROGRESS DURING FY 1994:**

During the year the following major accomplishments were achieved:

1. Several modifications to the 2.2 second drop tower apparatus were completed. The modifications include: 1) a new ignition system consisting of a hydrogen flame stabilized around the spherical burner then switching the fuel flow from hydrogen to the test fuel; 2) three thermocouples to measure the burner surface and two gas-phase temperatures; and 3) a novel flame radiation measurement consisting of three band-limited photodiodes, viz., 200-450nm, 200-1100nm, and 800-1800nm, to capture the shifting wavelength distribution expected during the transient reduced gravity flame.
2. Experimental observations in microgravity indicate that spherical flames can be obtained during the 2.2 second test time available in the drop tower, and that these flames evolve through large variations in radiative emissions as measured by the photodiode detectors. These measured emissions are to be compared to the theoretical model developed during previous years of this work.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 2

PhD Students: 0

TASK INITIATION: 4/91 EXPIRATION: 3/95

PROJECT IDENTIFICATION: 962-22-05-29

RESPONSIBLE CENTER: LeRC

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*Multicomponent Droplet Combustion in Microgravity: Soot Formation, Emulsions, Metal-Based Additives, and the Effect of Initial Droplet Diameter*

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PRINCIPAL INVESTIGATOR: Prof. C. T. Avedisian

Cornell University

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of the proposed research is to provide insights and data that supports the role of immiscible (e.g. emulsion) and miscible metal-containing additives, and to examine the influence of the initial droplet diameter on the combustion of fuel droplets in microgravity. In addition, the feasibility of using a laser-based light scattering technique for detecting soot formed in spherically symmetric droplet flames in microgravity, and imaging droplet flames in microgravity by coupling UV filtering with a conventional CCD camera that is coupled with an image intensifier, will be examined. Initially the experiments will be performed in a standard atmosphere.

**TASK DESCRIPTION:**

The burning of multicomponent droplets is complicated by the influence of composition, which can effect the formation of soot, lead to multistage combustion in which one component preferentially vaporizes from the droplet during combustion, or possibly result in the droplet exploding during combustion due to achieving an internal superheat condition. These problems are extremely complicated, especially that of soot formation. A CCD camera coupled with an image intensifier for the purpose of recording UV light emissions from the flame droplet will be measuring the droplet flame diameter and a laser light scattering technique will be set-up to provide a quantitative measure of soot formation. The experiments will be conducted in a drop tower at Cornell that provides approximately 1 second of reduced gravity.

**TASK SIGNIFICANCE:**

Little data have been provided regarding the spherically symmetric burning of unsupported droplets of multicomponent fuels or emulsions. The importance of examining fuel blends is that most practical fuels which are burned in combustion-powered devices or incinerators are multicomponent in nature, usually miscible, but sometimes immiscible (emulsions).

If the mechanism for an influence of initial droplet diameter on spherically symmetric droplet burning can be understood and predicted for the "simple" spherically symmetric droplet flame, this information may be useful for providing insights into the influence of initial droplet size on soot formation in a convective environment. Metal additives can create the potential for significant reductions in particulate emissions by the possible effect of the ions produced on the nucleation and agglomeration of soot particles, and their possible oxidation; their effect in the droplet flames, especially in the spherically symmetric configuration, is unknown.

**PROGRESS DURING FY 1994:**

There is no progress to report at this time since work will not start until May, 1995.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 5/95 EXPIRATION: 5/99

PROJECT IDENTIFICATION: 962-22-05-49

RESPONSIBLE CENTER: LeRC

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*Development of Advanced Diagnostics for Characterization of Burning Droplets in Microgravity*

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PRINCIPAL INVESTIGATOR: Dr. William D. Bachalo

Aerometrics, Inc.

CO-INVESTIGATORS:

Subra V. Sankar

Aerometrics, Inc.

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**TASK OBJECTIVE:**

This research is intended to develop rainbow thermometry for quantitative radial temperature measurements in burning droplets and use Morphologically-Dependent Resonances for quantification of the radial regression rate. Both of these techniques will be developed using devices amenable for use in one of NASA's reduced gravity aircraft; this hardware will be delivered to LeRC at the end of the project.

**TASK DESCRIPTION:**

Rainbow thermometry utilizes the angular dependence of rainbow location. In other words, given a known droplet composition, the location of the rainbow generated by that drop when illuminated with a monochromatic source will give the refractive index of the drop, and thus, its temperature. Instrumentation and software will be developed that will permit quantitative droplet temperature measurement.

Morphologically-Dependent Resonances occurs when the droplet diameter is an integral number of wavelengths of the illumination source. As the droplet size regresses due to combustion, this condition will be repeatedly met, causing the droplet to periodically "shine." The rate at which this occurs will give the diameter regression rate and will be accomplished using the same hardware as for the rainbow thermometry.

**TASK SIGNIFICANCE:**

Droplet combustion is an economically vital phenomenon occurring in liquid-fueled engine combustion, which includes both mobile and stationary combustors. Optimization of such designs has been hindered due to insufficient knowledge about the nature of combusting droplets. This project will develop analytic instrumentation that will allow measurement of the radial temperature distribution and size of combusting droplets. While the tools to be developed can be used in either normal or reduced gravity, the latter admits the use of much larger droplets and dramatically increases the information yield of the experiment.

**PROGRESS DURING FY 1994:**

The contract with Aerometrics was executed on August 1, 1994 and they were so informed on August 18, 1994. Since the initiation of the contract was late in the fiscal year, Aerometrics has only had time to form a Project Team and outline a work plan.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 8/94 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 962-22-05-43

RESPONSIBLE CENTER: LeRC

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### *Ignition and Combustion of Bulk Metals in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Melvyn C. Branch

University of Colorado, Boulder

#### CO-INVESTIGATORS:

Prof. J.W. Daily

University of Colorado, Boulder

G. J. Fiechtner

University of Colorado, Boulder

A. Abbud-Madrid

University of Colorado, Boulder

#### TASK OBJECTIVE:

This project is an investigation of the mechanisms of ignition and combustion of metals in bulk-pellet forms under low-pressure atmospheres over a range of gravity levels. The project extends the unique experimental features, database, and analytical models established in the previous normal-gravity study to the low-gravity environment.

#### TASK DESCRIPTION:

The preceding NASA-supported project devised an apparatus with a non-disturbing ignition source and dedicated diagnostics to determine surface and gas temperatures and metal-oxide surface morphology. A database of ignition delays and combustion behavior was obtained at normal gravity for a number of pure metal specimens. The present study extends the research to cover metal ignition and combustion behavior in both elevated and low gravity. Elevated-gravity measurements, already underway in the Geotechnical Centrifuge operated by the University of Colorado, are to be completed. The observation of the metal surface during ignition and combustion is possible through high-speed cinematography, using a camera on loan from the Lewis Research Center. Metal-oxide combustion specimens are recovered after each test and analyzed physically and chemically by electron microscopy. The compact, remotely operated apparatus used on the centrifuge will then be modified to meet the requirements and limitations for low-gravity experiments, to be conducted by the investigators in the Lewis Research Center airplane facility.

The analytical study involves numerical modeling of the metal heating, ignition, and combustion processes as influenced by gravity level and oxygen pressure. The initial findings were verified by the normal-gravity measurements of metal temperature rise, with excellent agreement. Extended modeling will incorporate the variations in heat loss by natural convection, to be validated by the elevated- and low-gravity measurements. The experimental results will also contribute information essential for the addition of surface and gas-phase reaction processes into the modeling.

#### TASK SIGNIFICANCE:

This study offers a novel means of observing and interpreting metal combustion using a radiant-energy ignition source and a simplified convection-controlled environment. The results offer new scientific information on metal combustion reactions, and they promise practical applications to the management and safety of spacecraft cryogenic propellant systems.

#### PROGRESS DURING FY 1994:

The quantitative measurement capabilities of the normal-gravity experimental study were extended to supplement the temperature measurements and visual observations obtained in the preceding year. Pure-metal specimens slightly smaller than before, 4 mm in diameter and 4 mm high, were heated by the unique radiant source of a 1000-W Xe short-arc lamp. The added diagnostics included a spectrograph-array detector system, scanning electron microscope (SEM) metallography, and X-ray spectrometry of specimens quenched at various stages during heat-up and combustion. In addition, surface-temperature histories indicative of the distinct stages of heat-up, ignition, and combustion were obtained over an elevated acceleration range of 2 to 20 g ( $g=9.8 \text{ m/s}^2$ ) in the University of Colorado Geotechnical Centrifuge.

The baseline screening tests were conducted with 8 different pure metals selected to exhibit a range of ignition and combustion behavior. The additional measurements were each conducted with a single metal for simplification, however.

The time-resolved measurements of gas-phase emission spectra in the reaction field were conducted with magnesium (Mg) specimens. A spectrograph with a diode-array detector gave a refined measurement of color temperature from the broad-band continuum spectrum. Prominent lines in the narrow-band spectra established the abundance of Mg and its MgO product through identified electronic and vibration resonances. This information will be very useful in determining the effect of gravity-induced convection in gas-phase reactions of bulk metals.

The SEM surface-morphology and chemical analyses of quenched specimens were conducted with copper (Cu) specimens. The oxidation sequence of Cu at various stages of the heating and combustion processes was determined from the identification and location of surface reactants and products, with further corroboration by quantitative electron microprobe analysis. It is anticipated that in microgravity the chemical nature of the oxide layer will be altered by the lack of convective transport of oxygen adjacent to the metal surface.

The elevated-gravity measurements were conducted with titanium (Ti) specimens. The complete experimental system was mounted on swiveling baskets at both ends of the rotating arms of the centrifuge. Power, control, and data signals were transmitted through slip rings. An additional silicon photodiode was installed to monitor any changes in the Xe arc lamp output due to the increased acceleration environment. Results of the testing, in terms of the time-related temperature responses, indicate a distinct change in the heating rate of the Ti samples. At 3 and 5g, the heating rate decreases and the ignition delay increases, although the ignition temperature remains comparable to that at normal gravity. At higher accelerations, however, the trend reverses and ignition delay decreases. In fact, at 15 and 20g, the heating rate is greater than at normal gravity and the ignition temperature appears to be higher. This behavior is apparently due to the competing effects of the increase in the transport rate of oxygen to the metal surface and the increase in convective heat loss with acceleration. This plausible mechanism will be confirmed in further experiments with a variety of metals and oxidizing atmospheres, along with more detailed spectroscopic diagnostics.

This project is to conclude early in FY 1995 (November 1994). Design activities are initiating to modify the apparatus for installation in an airplane facility, for a follow-on microgravity project already approved for support by NASA Headquarters.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 4/91 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 962-22-05-30

NASA CONTRACT NO.: NAG3-1257

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Abbud-Madrid, A., Fiechtner, G.J., Branch, M.C., and Daily, J.W. "A study of bulk metal ignition in oxygen atmospheres." The Combustion Institute, Fall Meeting of the Western States Section, WSS/CI 93-079, October 18-19, 1993.

#### Presentations

Abbud-Madrid, A., Fiechtner, G.J., Branch, M.C., and Daily, J.W. "Ignition and combustion characteristics of pure bulk metals: Normal gravity test results." AIAA Paper 94-0574, 32nd Aerospace Sciences Meeting and Exhibit, Reno, NV, January 10-13, 1994.

### *Ignition and Combustion of Bulk Metals in Microgravity (Ground-Based Experiment)*

PRINCIPAL INVESTIGATOR: Prof. Melvyn C. Branch

University of Colorado, Boulder

#### CO-INVESTIGATORS:

Prof. J.W. Daily  
A. Abbud-Madrid

University of Colorado, Boulder  
University of Colorado, Boulder

#### TASK OBJECTIVE:

This project is an investigation of the mechanisms of ignition and combustion of metals in bulk pellet forms under low-pressure atmospheres over a range of gravity levels. The project extends the unique experimental features, database, and analytical models established in the previous normal gravity study to the low gravity environment.

#### TASK DESCRIPTION:

The preceding NASA supported project devised an apparatus with a non-disturbing ignition source and dedicated diagnostics to determine surface and gas temperatures and metal oxide surface morphology. A database of ignition delays and combustion behavior was obtained at normal gravity for a number of pure metal specimens. The present study extends the research to cover metal ignition and combustion behavior in both elevated and low gravity. Elevated gravity measurements, already underway in the Geotechnical Centrifuge operated by the University of Colorado, are to be completed. The observation of the metal surface during ignition and combustion is possible through high-speed cinematography, using a camera on loan from the Lewis Research Center. Metal oxide combustion specimens are recovered after each test and analyzed physically and chemically by electron microscopy. The compact, remotely operated apparatus used on the centrifuge will then be modified to meet the requirements and limitations for low gravity experiments, to be conducted by the investigators in the Lewis Research Center airplane facility.

The analytical study involves numerical modeling of the metal heating, ignition, and combustion processes as influenced by gravity level and oxygen pressure. The initial findings were verified by the normal gravity measurements of metal temperature rise, with excellent agreement. Extended modeling will incorporate the variations in heat loss by natural convection, to be validated by the elevated and low gravity measurements. The experimental results will also contribute information essential for the addition of surface and gas-phase reaction processes into the modeling.

#### TASK SIGNIFICANCE:

This study offers a novel means of observing and interpreting metal combustion using a radiant-energy ignition source and a simplified convection controlled environment. The results offer new scientific information on metal combustion reactions, and they promise practical applications to the management and safety of spacecraft cryogenic propellant systems.

#### PROGRESS DURING FY 1994:

This project is not scheduled for initiation until December 1994, upon completion of the current normal and elevated gravity project.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 12/94 EXPIRATION: 11/97

PROJECT IDENTIFICATION: 962-22-05-30

RESPONSIBLE CENTER: LeRC



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*Modeling of Microgravity Combustion Experiments*

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PRINCIPAL INVESTIGATOR: Prof. John D. Buckmaster

University of Illinois, Urbana-Champaign

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

Modeling of existing microgravity experiments for improving our understanding of the fundamental physics, with particular emphasis on (a) premixed gaseous combustion processes, particularly those involving purely diffusive and very slow convective velocities, and (b) chattering flame propagation in premixed particle cloud combustion.

**TASK DESCRIPTION:**

Microgravity combustion experiments amenable to modeling are identified, and analytical and numerical models are developed from first principles for various systems and configurations. Model predictions are compared with experimental results and necessary adjustments and enhancements are incorporated into the models. Precise quantitative comparisons are made with numerical results where possible.

**TASK SIGNIFICANCE:**

Modeling provides invaluable physical insights into the experimentally observed behavior. This study is done in close collaboration with experimentalists with the goal of providing a clear understanding of the underlying physics.

**PROGRESS DURING FY 1994:**

A presentation entitled "Unsteady Spherical Flames Generated by Point Ignition of Combustible Mixtures Containing Inert Particles," by J. Buckmaster and A. Agarwal was given by J. Buckmaster at the 5th Int. Conf. on Numerical Combustion in Garmisch, Sept. 27 - Oct. 1, 1993.

A poster session presentation entitled "Unsteady Spherical Flames in Dusty Gases," by J. Buckmaster and A. Agarwal was given at the 25th International Symposium on Combustion, Irvine, CA, 1994. It is also submitted to a journal as a paper for publication. The abstract of the paper reads: "We examine an expanding spherical premixed flame propagating in a mixture containing inert dust. The presence of the dust generates a significant radiative energy flux that influences the flame speed. With the adoption of a hydrodynamic description in which stretch effects are accounted for using an empirical formula, and with the use of the Eddington approximation to describe the radiation, a simple numerical problem is formulated valid for that initial time interval in which the flame temperature changes by  $O(\epsilon)$  amounts where  $\epsilon$  is the small inverse activation energy. Solutions are constructed using parameter values appropriate to lean  $\text{CH}_4$ /air mixtures, and for modest particle loadings. We describe the competition between the Zeldovich-Spalding effect in which radiative losses on the diffusive scale tend to quench the flame, and the Joulin effect in which radiative preheating on the scale of the Planck length tends to strengthen the flame."

The paper entitled "The role of slip in the generation of acoustic instabilities in gas turbine combustion systems," by M. DiCicco and J. Buckmaster was completed in December 1993 and submitted for publication. The abstract of the paper reads: "In the context of gas turbine combustion chambers, this study describes how slip affects the response time of fuel sprays to pressure fluctuations in a gaseous flow field. Slip between the condensed and gas phases is shown to cause fuel vapor mass fraction fluctuations upstream of the reaction zone. A resulting oscillating heat release can drive the pressure fluctuations, depending on the phase difference between them. This generates an acoustic instability. With relevance to previous experimental results, differences are explored in the evaporation characteristics among three different fuel sprays (JP-4, JP-5, and D-2) in relation to their effect on the magnitude of the fuel vapor mass fraction perturbations. Parameters such as droplet sizes, inlet air temperatures, air velocities, and initial Reynolds numbers, are varied with their corresponding influence examined."

The paper entitled "Acoustic instabilities driven by slip between a condensed phase and the gas phase in combustion systems," by M. DiCicco and J. Buckmaster was presented by M. DiCicco (graduate student) at the 32nd AIAA Aerospace Sciences Meeting, Reno, Nevada, in January 1994. The abstract of the paper reads: "In the context of gas turbine combustion chambers, this study describes how slip affects the response time of fuel sprays to pressure fluctuations in a gaseous flow field. Slip between the condensed and gas phases is shown to cause the fuel vapor mass fraction fluctuations upstream of the reaction zone. A resulting oscillating heat release can drive the pressure fluctuations, depending on the phase difference between them. This generates an acoustic instability. With relevance to previous experimental results, differences are explored in the evaporation characteristics among three different fuel sprays (JP-4, JP-5, and D-2) in relation to their effect on the magnitude of the fuel vapor mass fraction perturbations."

The paper entitled "Intrinsic and acoustic instabilities in flames fueled by multiphase mixtures," by C.J. Lee, J. Buckmaster, and M. DiCicco was published in Combustion Science and Technology in 1994. The abstract of the paper reads: "We describe linear stability results for flames fueled by mixtures of air and particles. Nonsimilarity between the temperature and fuel concentration fields gives rise to an intrinsic pulsating instability for unconfined flames (a single mode). Acoustic interactions for confined propagating flames generate acoustic instabilities (an infinite number of modes) when the gas-phase velocity is different from that of the solid phase (i.e. there is slip). The confinement modes can not, in general, be classified as intrinsic, fundamental, first-harmonic, etc. For example, for propagation in a tube with flame initiation at the open end, a mode can start as the second harmonic but finish, when the flame is at the closed end, as the fundamental. Instabilities tend to be suppressed in spherical flames generated by point ignition in a confinement vessel. The triggering of acoustic instabilities in gas turbines by slip between fuel drops and air is discussed, and we show that the role of slip is quite different when the condensed phase is injected at a finite point rather than being dispersed throughout the gas phase."

The paper entitled "The effects of radiation on the thermal-diffusive stability boundaries of premixed flames," by J. Buckmaster and T. Jackson was accepted for publication in April 1994 in Combustion Science and Technology. The abstract of the paper reads: "We examine the stability of premixed flames in mixtures containing significant amounts of fine dust whose sole impact is upon the radiative transport. By using well-established modeling strategies together with a simple radiation model which preserves much of the essential physics, it is possible to explore to what extent radiative transport displaces the classical non-hydrodynamical stability boundaries of the plane deflagration. Analysis is possible for arbitrary values of both the Planck length and the Boltzman number. It is shown that the pulsating/traveling-wave instability is strongly enhanced by the presence of radiation, and can be present even if  $Le < 1$ . On the other hand, radiation tends to suppress the cellular instability normally associated with values of  $Le$  less than 1. The latter is consistent with preliminary experimental observations of Abbud-Madrid and Ronney."

The paper entitled "Absolute flammability limits and flame-balls," by D. Lozinski and J. Buckmaster was published in Combustion and Flame in 1994. The abstract of the paper reads: "We examine flame balls that are not optically thin so that radiation from the hot burned gas is not lost but is reabsorbed by the surrounding cool gas. The steady solution is shown to be multivalued in general and defines an intrinsic (apparatus independent) flammability limit for a sufficiently small ignition source. Beyond the flammability limit a steady solution exists for arbitrarily weak mixtures, but is unrealizable since it is unstable to one-dimensional perturbations. An unsteady propagating flame solution is also possible for arbitrarily weak mixtures, but can only be generated by a large ignition source. Observations of flame-strings in SF<sub>6</sub>-diluted mixtures are reported and suggest that the peculiar nature of the radiative properties of SF<sub>6</sub> is responsible for peculiar dynamics."

The paper entitled "Quenching of opposed forced flow smoulder," by D. Lozinski and J. Buckmaster was presented by D. Lozinski, post-doctoral associate, at the Spring Meeting of the Canadian Section of the Combustion Institute in May 1994. This work was subsequently completed as a paper entitled "Quenching of flames in reverse smoulder," by D. Lozinski and J. Buckmaster in June 1994 and submitted for publication. The abstract of the paper reads: "A simple model of reverse smoulder in a porous medium is analyzed using asymptotic methods. When the only chemical reaction is exothermic oxidation, the burning rate is a single-valued function of the blowing rate, increasing from zero to a maximum, and then returning to zero. When endothermic pyrolysis is added to the description, the burning rate is double-valued for blowing rates less than some maximum. Beyond this maximum

there are no solutions. The upper branch of the double-valued solution is the physically relevant one. On it, for certain choices of parameters, the burning rate increases from zero to a maximum, and then decreases until quenching occurs at the maximum blowing rate. This behavior mimics experimental observations by Torero, Fernandez-Pello, and Kitano (1993)."

The paper entitled "The role of mathematical modeling in combustion," by J. Buckmaster was published in a book entitled *Combustion in High Speed Flows*, J. Buckmaster, T. Jackson, and A. Kumar, eds., Kluwer Academic Publishers, The Netherlands, 1994, pp. 447-459. (S. Gokoglu)

The book entitled *Combustion in High Speed Flows* was published, J. Buckmaster, T. Jackson, and A. Kumar editors, by Kluwer Academic Publishers, The Netherlands, in 1994.

The paper entitled "Some topics in reverse smoulder," by J. Buckmaster and D. Lozinski, based on a presentation made by J. Buckmaster at a US/Japanese Workshop on Modeling in Combustion, held in Kauai, July 1994, was completed and submitted in August 1994 for the Workshop Proceedings. The abstract of the paper reads: "The role of thermal non-equilibrium and endothermic pyrolysis is discussed in the context of a simple model of reverse smoulder combustion. It is shown that non-equilibrium has little qualitative effect on the nature of the solutions, but endothermic pyrolysis can lead to quenching at sufficiently large blowing rates."

The paper entitled "An elementary discussion of forward smoldering," by J. Buckmaster, C. Fernandez-Pello, and D. Lozinski is submitted for publication. The abstract of the paper reads: "We describe an elementary model of one-dimensional unsteady forward smoldering, purged of all unnecessary physics. Following work of Dosanjh and Pagni, a late time solution is constructed, characterized by two reaction fronts - an exothermic oxidation front, an unusual kind of diffusion flame; and an endothermic pyrolysis front. It is shown that the flame temperature and the ratio of the speeds of the two fronts relative to the solid are independent of the blowing rate, in agreement with data obtained by Ohlemiller and Lucca. The structure of the oxidation front is described in the context of 1-step Arrhenius kinetics, and it is shown that leakage of solid reactant through the front is possible, but not leakage of oxygen. An elementary pyrolysis structure is also examined which reduces to the frontal model in a certain limit, and clarifies its nature."

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	3	MS Degrees:	2
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 2/91 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-22-05-31

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Buckmaster, J. The structure and stability of laminar flames. *Annu. Rev. Fluid Mech.* vol. 25, 21-53 (1993).

Buckmaster, J. The role of mathematical modeling in combustion. *Combustion in High Speed Flows*, Kluwer Academic Publishers, The Netherlands, 447-459 (1994).

Buckmaster, J. and G. Joulin *Comb. Sci. & Tech.*, vol. 89, 57-69 (1993).

Buckmaster, J. and Jackson, T. The effects of radiation on the thermal-diffusive stability boundaries of premixed flames. accepted by *Combustion Science and Technology*, (1994).

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DiCicco, M. and Buckmaster, J. Acoustic instabilities driven by slip between a condensed phase and the gas phase in combustion systems. *AIAA*, 94-0103, (1994).

Lee, C.J., Buckmaster, J. and DiCicco, M. Intrinsic and acoustic instabilities in flames fueled by multiphase mixtures. Comb. Sci. & Tech. vol. 98, 161 (1994).

Lozinski, D. and Buckmaster, J. Absolute flammability limits and flame-balls. Comb. & Flame. vol. 97, 301-316 (1994).

### Presentations

Buckmaster, J. and A. Agarwal "Unsteady spherical flames generated by point ignition of combustible mixtures containing inert particles." presented at the 5th Int. Conf. on Numerical Combustion in Garmisch, September 27 - October 1, 1993.

Buckmaster, J. and A. Agarwal ""Unsteady spherical flames in dusty gases"" presented at the 25th Int. Symp. on Combustion, The Combustion Institute, Irvine, CA, July 31 - August 5, 1994.

Buckmaster, J. and Agarwal, A. "Unsteady spherical flames in dusty gases." poster session presentation at the 25th Int. Symp. on Combustion, Irvine, CA, July 31 - August 4, 1994.

Buckmaster, J. and DiCicco, M. "Acoustic instabilities driven by slip between a condensed phase and the gas phase in combustion systems." presented at the AIAA 32nd Aerospace Sciences Meeting, Reno, NV, Jan. 10-13, 1994.

Buckmaster, J. and Jackson, T. "The effect of radiative transport on the thermal-diffusive stability boundaries of premixed flames." poster session presentation at the 25th Int. Symp. on Combustion, Irvine, CA, July 31 - August 4, 1994.

Buckmaster, J. and Lozinski, D. "Some topics in revedrse smoulder." presented at the US/Japanese Workshop on Modeling in Combustion, Kauai, July, 1994.

Lozinski, D. and Buckmaster, J. "Quenching of opposed forced flow smoulder." presented at the 1994 Spring Technical Meeting of the Canadian Section of the Combustion Institute, 10-13 May, 1994.

Lozinski, D. and J. Buckmaster "Quenching of opposed forced flow smoldering combustion." presented at the 1994 Spring Technical Meeting of the Canadian Section of the Combustion Institute, May 10-13, 1994.

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*Buoyancy Effects on the Structure and Stability of Burke-Schumann Diffusion Flames*

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PRINCIPAL INVESTIGATOR: Prof. L. D. Chen

University of Iowa

## CO-INVESTIGATORS:

Dennis P. Stocker

NASA Lewis Research Center (LeRC)

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TASK OBJECTIVE:

The general goals of the proposed research are to improve our understanding of (1) the influence of buoyancy on co-flow diffusion flames, e.g., Burke-Schumann diffusion flames, and (2) the effects of buoyancy on vortex-flame interactions in co-flow diffusion flames. The overall objectives of the proposed work are to:

1. Verify the zero-gravity Burke-Schumann model and the gravity-dependent Hegde-Bahadori extension.
2. Investigate the flame stability in a buoyancy-dependent flow-field as affected by the co-flow oxidizer.
3. Examine the state relationships of co-flow diffusion flames.
4. Study flow vortex and diffusion flame interactions.

## TASK DESCRIPTION:

In order to meet the objectives, the phenomena will be investigated by:

1. Microgravity testing.
2. Normal-gravity testing (e.g., at reduced pressure to emulate reduced gravity).
3. Numerical modeling.

D. Stocker (NASA LeRC) will have the lead responsibility for the microgravity testing, which will be generally conducted in the 2.2-second Drop Tower. The tests will be conducted in the rig fabricated for Stocker's precursor studies of Burke-Schumann diffusion flames. Also, D. Stocker and L.-D. Chen will consider testing in the Zero-G Facility and in research aircraft (e.g., DC-9) if a need is identified. In either case, the tests will most likely be conducted in rig(s) built for the Gas-jet Diffusion Flame (GDF) Experiment, since only limited modification would be required.

Normal-gravity tests will be conducted under the direction of Prof. Chen at the University of Iowa and possibly at the Wright Laboratory (at WPAFB, in Dayton, OH), since the WPAFB facilities have been made available to this project at no cost. The normal-gravity experiments will complement the microgravity experiments, but will include tests that cannot be readily conducted in the microgravity facilities. For example, some instrumentation (e.g., NO<sub>x</sub> analyzer) that will be used in the normal-gravity testing cannot readily be used in the microgravity tests due to various constraints (e.g., time required for measurement, size of instrument, "ruggedness" of instrument, etc.).

Prof. Chen will direct the numerical modeling which will be conducted at the University of Iowa. An existing, semi-implicit, transient, axisymmetric code will be modified to include gas-phase radiation, state relationships or reduced mechanisms for major species, and finite chemistry for NO<sub>x</sub>. The predictions will be verified by the experimental measurements.

## TASK SIGNIFICANCE:

The 1928 Burke-Schumann diffusion flame analysis is of fundamental and historical interest, and can be found in many, if not all, introductory combustion textbooks in the U.S. Experimental validation of this zero-gravity model will be an important contribution to combustion science, and it is likely to be referenced in future textbooks. Additionally, the study results could lead in the long-term to improved engine or furnace efficiency and reduced pollutant emissions.

### PROGRESS DURING FY 1994:

During FY94, the work was focused on the development for upcoming tests and the modeling and analysis of previous results. There were no microgravity tests conducted in FY94, due to the shut down of the 2.2-second Drop Tower. The analysis of the pulsed Burke-Schumann flame tests conducted in FY93 was presented at the WSS/CI Fall 1993 meeting and in a poster session at the Twenty-fifth International Symposium on Combustion, in August 1994.

#### Hardware Development

Whereas the previous microgravity tests have been limited to photographic studies of the flame structure, the upcoming tests (to begin in FY95) will include measurements of temperature, species, etc. For this reason, a significant effort was focused on the hardware preparation for the upcoming microgravity tests.

In the fall of 1993, D. Stocker was assisted in this effort by two undergraduate interns receiving design course credit from Worcester Polytechnic Institute. Flow visualization in the co-flow of the Burke-Schumann burner was demonstrated using streak imaging, by seeding the air flow with phenolic microballoons or smoke (from model-train smoke fluid). Shadowgraph imaging (using an expanded beam from a 15-mW HeNe laser) was used to visualize the hot plume above the flame. Additionally, tests were conducted with various slot burners in preparation for the design of slot burner(s) to be included in upcoming tests. Slot flames are of interest, since the analysis of Roper and Hegde & Bahadori predicts that they will be more strongly dependent on buoyancy than the axisymmetric flames studied by Stocker thus far.

J. Goldmeer, a LeRC GSRP fellow, and D. Stocker conducted preliminary normal-gravity tests using thin filament pyrometry (TFP) to measure temperature profiles across a flame. Multiple fibers were imaged with a standard video camera, via an interference filter, providing a strong intensity and low noise with the camera at maximum gain. Intensity profiles along each fiber are easily obtained using the Image Processing workstation. Calibration of the video intensity vs. flame temperature will be done in FY95. TFP will be the primary method for temperature measurement in the upcoming drop tests.

LeRC provided a driver for a proportional control valve to produce sinusoidal variations in the fuel flow for pulsed flame studies. The droppable device sinusoidally varies the flow, and provides (nearly) independent control of the frequency and amplitude (both dc and ac) of the flow. Although there may be some hysteresis in the flow, the preliminary tests are promising. The periodic flow from the device will be characterized in FY95.

Work is in progress on the definition of requirements and the conceptual design of circular and slot Burke-Schumann burners, which will be appropriate for smoke-fluid flow visualization, thin-filament pyrometry, absorption spectroscopy for species measurements, and traditional instrumentation (e.g., thermocouples). For the species measurements, a new source/detector array will be designed, that will mate with the burner chimney and be used with the spectroscopy system developed by Southwest Sciences, Inc. The burners will include interchangeable chimney segments appropriate to the different measurement techniques.

#### Modeling & Analysis

Stocker and the two WPI interns coded a computer program for predictions of the height and shape of slot flames for three conditions: (1) no gravity and no axial diffusion, (2) with gravity but no axial diffusion, and (3) no gravity but with axial diffusion, based on the works of (1) Burke & Schumann, (2) Chung & Law, as well as Roper, and (3) Hegde & Bahadori.

Modeling was also performed at the University of Iowa for comparison with the pulsed flame tests conducted by Stocker in FY93. A correlation was established between the tip cutting and the appearance of flow vortices. The numerical modeling showed that the tip cutting coincided with vortex formation near the tip of the flame in 1g, and near the base of the flame in 0g. Furthermore, animation of the vorticity field has shown that the baroclinic effect in the fuel-lean region acts as a source term in vortex dynamics, while thermal expansion is a sink term.

The modeling was unable to predict, however, observed subharmonic flame response, which presumably results from vortex merging. This held true despite the use of FCT correction for convective transport, various velocity profiles, and various waveforms in the modeling. Based on these results, differential pressure measurements were made of the flow pulsation system, but they revealed a fundamental response, ruling out the solenoid valve as the cause of the subharmonic response observed in the flames.

Nonetheless, numerical simulation of a flame pulsed at 1 Hz has shown the effect of buoyancy on the flame's spectral response. The normal-gravity flame spectrum appears to consist of linear combinations of two dominant frequencies, 1 and 4 Hz. The microgravity flame spectrum showed only one dominant frequency, the 1-Hz forcing frequency.

Modeling and normal-gravity experiments at University Iowa revealed a harmonic response (in flame height and temperature traces) in Burke-Schumann flames pulsed at 1 to 4 Hz. Thus results thus far show that buoyancy is an important parameter in this nonlinear flame response (as suggested by Stocker's drop tests). A paper is being prepared describing the numerical algorithm, that will use this finding as an example of the importance of using a high-order scheme (spatial) for accurately predicting flame dynamics.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 2

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TASK INITIATION: 6/94 EXPIRATION: 6/98

PROJECT IDENTIFICATION: 962-22-05-63

RESPONSIBLE CENTER: LeRC

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*Gravitational Effects on Premixed Turbulent Flames*

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PRINCIPAL INVESTIGATOR: Dr. Robert K. Cheng

Lawrence Berkeley Laboratory

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The overall objectives of this experiment is to investigate experimentally the dynamics of low Reynolds number premixed turbulent flames in a microgravity environment.

**TASK DESCRIPTION:**

The emphasis of this experimental program will be on measuring flame wrinkle scales and imaging mean flowfield properties of conical Bunsen-type flames and rod-stabilized v-flames. Laser diagnostics will be used to obtain statistical scalar and velocity information to evaluate parameters suitable for predicting the effects of gravity on turbulent premixed flames. A 2.2 second drop tower, and Learjet parabolas will be performed as well as parallel laboratory experiments.

**TASK SIGNIFICANCE:**

This research effort will provide insight to determine significant processes through which gravity affects flame properties such as flame speed and flame structures. The results will be used to determine the gravity-influenced limit for premixed turbulent flames. Such knowledge is valuable for guiding the development of turbulent combustion models to include the effects of gravity. This experimental study will contribute to resolving the inconsistencies that exist between experiments and theories and help couple chemical and fluid mechanical processes found in many turbulent combustion environments. If better understood, turbulent combustion can be exploited to enhance burning rates and volumetric power density in many heating and power generating systems.

**PROGRESS DURING FY 1994:**

An experimental rig that had been used in the 2.2-second Drop Tower was converted for the Lear jet. The behavior of laminar and turbulent Bunsen type conical and rod stabilized v-flames has been observed by the use of laser schlieren. The tests are follow-on experiments based on 2.2 second drop tower work from previous years. The schlieren images are recorded on videotape and are analyzed by computer-controlled image processing. Comparison of the microgravity results with those under normal gravity shows the gravity affects flame propagation through the coupling of the flame dynamics and the surrounding flowfield. Under normal gravity, buoyancy-driven flow instabilities are shown to induce flame flickering.

The emphasis of the microgravity experiment was on measuring mean flame properties and compared them with those observed in the laboratory under normal gravity and reversed gravity. In addition to the use of laser schlieren, the laboratory flames are also interrogated by other laser technique to measure their scalar and velocity statistics. These information are needed to evaluate parameters suitable for predicting the effects of gravity on premixed flames. The data will be useful for comparisons to theoretical models which do not include the effects of gravity. The study will assist in understanding the relationships between turbulence and low-gravity combustion.

The analysis of the schlieren video during the Learjet experiments has begun. For the laminar flame cases, the median of the intensity is extracted to represent the mean flame position. For the turbulent flame cases, the task was more difficult due to the broad flame position. Different methods are being explored to obtain consistent and reproducible representation of the turbulent flames. The analysis of gravity-driven pulsating frequency of the Bunsen flame is complete. An empirical correlation is found for all the pulsating frequencies measured in our laboratories and those reported previously by others. The independent parameter is the square of Strouhal number over the Richardson number. The dependent parameter is the Reynolds number to the power of 2/3. The influence of gravity on the stabilization limits of Bunsen flames and rod-stabilized v-flames are also determined. These results



are useful for designing future microgravity experiment and to elucidate the processes important to characterizing the coupling between gravity and premixed flames.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
 MS Students: 0  
 PhD Students: 1

**TASK INITIATION:** 6/91 **EXPIRATION:** 3/95

**PROJECT IDENTIFICATION:** 962-22-05-32

**RESPONSIBLE CENTER:** LeRC

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*Gravitational Effects on Premixed Turbulent Flames: Microgravity Flame Structures*

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PRINCIPAL INVESTIGATOR: Dr. Robert K. Cheng

Lawrence Berkeley Laboratory

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The overall objectives of this experiment is to investigate the influence of gravity on premixed turbulent flame propagation.

**TASK DESCRIPTION:**

The emphasis of this experimental program will be on measuring flame wrinkle scales and imaging mean flowfield properties of conical Bunsen-type flames and rod-stabilized V-flames. Laser diagnostics will be used to obtain statistical scalar and velocity information to evaluate parameters suitable for predicting the effects of gravity on turbulent premixed flames. Learjet parabolic flights will be performed as well as parallel laboratory experiments.

**TASK SIGNIFICANCE:**

This research effort will provide insight to determine significant processes through which gravity effects flame properties such as flame speed and flame structures. The results will be used to determine the gravity-influenced limit for premixed turbulent flames. Such knowledge is valuable for guiding the development of turbulent combustion models to include the effects of gravity. This experimental study will contribute to resolving the inconsistencies that exist between experiments and theories and help couple chemical and fluid mechanical processes found in many turbulent combustion environments. If better understood, turbulent combustion can be exploited to enhance burning rates and volumetric power density in many heating and power generating systems.

**PROGRESS DURING FY 1994:**

There is no progress to report at this time since work will not begin until March, 1995.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 3/95 EXPIRATION: 3/97

PROJECT IDENTIFICATION: 962-22-00

RESPONSIBLE CENTER: LeRC

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*Combustion of Interacting Droplet Arrays in a Microgravity Environment*

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PRINCIPAL INVESTIGATOR: Dr. Daniel L. DietrichNYMA, Inc.

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## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

This research program involves the study of one-dimensional and two-dimensional arrays of droplets in a buoyant-free environment. The purpose of the work is to extend the data base and theories that exist for single droplets into the regime wherein droplet interactions are important.

**TASK DESCRIPTION:**

The emphasis of the present investigation is experimental, although comparison will be made to existing theoretical and numerical treatments when appropriate. Both normal-gravity and low-gravity testing will be employed, and the results compared.

The normal-gravity testing will utilize the classical suspended droplet technique; single droplets and droplet arrays will be supported on 125  $\mu\text{m}$  optical fibers in a combustion chamber where the ambient environment can be controlled. The low-gravity testing will employ droplets suspended on 15  $\mu\text{m}$  Si-C fibers, a new technique developed during the past year, again in a combustion chamber where the ambient environment can be changed.

**TASK SIGNIFICANCE:**

The eventual goal will be to use the results of this work as inputs for models on spray combustion, wherein droplets seldom burn individually (the combustion history of a droplet is strongly influenced by the presence of the neighboring droplets).

**PROGRESS DURING FY 1994:**

In collaboration with Dr. R. Vander Wal, data was obtained on the relative and absolute soot volume fractions produced during the burning of fiber supported droplets. The data was of single droplets with a 1.5 mm initial diameter suspended on optical fibers at 1 atm pressure in the air. The fuels used were n-heptane, n-decane, chloroheptane, chlorodecane and ethanol/decane mixtures. The data consisted of simultaneous measurements of the soot volume fraction (via LII) and natural flame images, simultaneous measurements of natural flame and OH chemiluminescence, and simultaneous measurements of droplet size, flame radiation measurements, and natural flame.

The modifications to the experimental apparatus were completed and initial burning rate data on single droplets of pure fuels (decane, heptane, and methanol) with different size support fibers were taken. The effects of the support fiber are being investigated by comparing the experimental results to theoretical computations by M. Vedha-Nayagam.

The normal gravity apparatus was reconfigured for two droplet studies. Testing began on the combustion of two droplets of n-decane (normal gravity).

This effort is also part of the Fiber Supported Droplet Combustion Glovebox Experiment. The engineering model of the experiment was built and tested. The flight hardware was designed and all of the parts were fabricated. All of the paperwork to date is complete. The experiment is manifested on USML-2.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/89    EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-22-05-33

RESPONSIBLE CENTER: LeRC

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BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Presentations**

Dietrich, D. "Relative soot volume fractions in droplet combustion via laser-induced incandescence." Eastern States Section of the Combustion Institute Fall meeting, 1994.

Vander Wal, R. and Dietrich, D. "Laser-induced incandescence applied to droplet combustion." Applied Optics, 1994.

Vander Wal, R. and Dietrich, D. "Laser-induced incandescence applied droplet combustion." Western States Section of the Combustion Institute, Spring Meeting, 1994.

*Internal and Surface Phenomena in Heterogeneous Metal Combustion*

PRINCIPAL INVESTIGATOR: Dr. Edward L. Dreizin

AeroChem Research Laboratories, Inc.

CO-INVESTIGATORS:

Dr. H.F. Calcote

AeroChem Research Laboratories, Inc.

**TASK OBJECTIVE:**

This project is an experimental and analytical study to determine the mechanisms by which oxygen penetrates within a burning metal droplet and affects the combustion process. Particular attention is given to disruptive burning, such as in micro explosions, which have been observed but never explained by current theories.

**TASK DESCRIPTION:**

The study uses a novel apparatus that generates uniformly sized metal droplets through electrical arc discharges with consumable, pure metal electrodes. The separated droplets are ignited and then subsequently quenched to terminate the reaction at prescribed times during combustion. Experimental measurements will cover droplet temperatures and diameters as functions of time and chemical and metallurgical analyses of the quenched, solidified specimens. Test conditions include variations of temperature, droplet size, and atmospheric oxygen concentration, for at least three different metals. Initial normal gravity tests are conducted at the AeroChem Laboratories facilities. Subsequent microgravity tests, using a modified version of the droplet generator apparatus, will be conducted by the investigators in the Lewis Research Center free-fall drop tower facility. In microgravity, the near-absence of settling forces permits the precise control of the droplet motion to improve the interpretation of the droplet-diameter and droplet-temperature histories. Concurrent with the experimental studies, analytical studies verified by the experimental results model the processes occurring in metal droplet combustion to provide a theoretical foundation of surface and internal transport and reaction rate phenomena in the burning metal-droplet system.

**TASK SIGNIFICANCE:**

This project is a study of metal and oxygen behavior in metal-droplet combustion using a unique but demonstrated apparatus to generate, control, and analyze the combustion processes. The results of the study have practical application to the performance and improvement of metallized fuels, such as those used in solid rocket motors.

**PROGRESS DURING FY 1994:**

This project began on July 6, 1994 with the award of NASA Contract NAS 3-27259. Since the basic apparatus for metal-droplet generation and ignition, called the GEMMED, was available and operational, only minor modifications to the apparatus were required prior to the studies. These changes, already accomplished, included an improved consumable wire-electrode feed unit with a measuring microscope and various small changes to the power, gas-feed, and instrumentation systems.

The initial study used the GEMMED to generate aluminum (Al) droplets in three nominal diameters of 120  $\mu\text{m}$ , 160  $\mu\text{m}$ , and 190  $\mu\text{m}$ , for ignition and combustion in a normal-gravity, air environment. Temperature histories were provided by three-wavelength optical pyrometry. Three stages of combustion were observed in the available time for particle combustion (100 ms for the 160  $\mu\text{m}$  particle diameter, for example). 1) An initial stage had spherically symmetric combustion, with temperatures increasing to near the boiling point of  $\text{Al}_2\text{O}_3$  and radiation also increasing. 2) A second stage had non-symmetric combustion, with temperatures falling slightly but radiation showing a peak and oscillations. 3) A final stage had non-symmetric combustion, with decreasing temperatures and radiation.

Particles were quenched at different combustion times in the tests by allowing them to pass into an argon atmosphere separated from the air by a soap-bubble film as well as by impingement. Cross-sections of quenched

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

particles were examined using scanning electron microscopy; internal composition was determined using energy-dispersing spectroscopy. From the temperatures, composition, and observed smoke traces, the investigators propose a mechanism for the combustion-stage transition based on the concentration of oxygen dissolved in the Al particles. Further interpretations are being advanced to understand the nature of the final combustion stage and the potential to create micro-explosions.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/94 EXPIRATION: 6/98

PROJECT IDENTIFICATION: 962-22-05-46

NASA CONTRACT NONAS-3-27259

RESPONSIBLE CENTER: LeRC

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*Flame-Vortex Interactions Imaged in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. James F. Driscoll

University of Michigan

CO-INVESTIGATORS:

Prof. W.J.A. Dahm

University of Michigan

Prof. M. Sichel

University of Michigan

**TASK OBJECTIVE:**

The objective of this research is to investigate a method that will provide high quality, quantitative, color enhanced digital images of a vortex exerting aerodynamic strain on a flame under microgravity conditions. This will be used to:

1. Quantify how the vortex distorts the flame;
2. Define the degree of flame curvature;
3. Show how preferential diffusion affects temperature profiles.

**TASK DESCRIPTION:**

The fundamental interaction between turbulence and chemistry in a combustion process will be studied by interacting a repeatable, well-defined vortex, or variable size and strength vortex, with premixed and nonpremixed flames. The complicating effects of buoyancy will be eliminated by employing the NASA LeRC 2.2-second drop tower. High quality, quantitative, color enhanced 2-D images will be obtained of the temperature field, the flame emission, and the mixture fraction (nonpremixed case). Results will be compared to full Navier-Stokes direct numerical simulations and to the classical theory of stretched flames.

**TASK SIGNIFICANCE:**

Images of the flame shape and curvature will be used to assess numerical simulations which neglect buoyancy, and to deduce universal, buoyancy free scaling relations showing the effects of vortex size and strength. Understanding of turbulent combustion, applicable to all practical combustion, will be enhanced.

**PROGRESS DURING FY 1994:**

During the month of June a student was hired and was assigned the task of building and testing an array of Thin Filament Pyrometers, which is one of the diagnostics to be used on the drop tower experiment. A method was developed to mount 50 SiC filaments for thin filament pyrometry with the proper tension. The necessary flow metering equipment for the calibration burner was purchased and assembled. An intensified CCD array camera is being used to image the radiated light intensity from the 16 micron diameter filaments at two wavelengths, from which temperature is to be deduced. Also, during the month of June some of the necessary software was written and an orientation visit from Jim Driscoll, Werner Dahm, Chuck Mussel, and Ron Springer, took place on 24 June 1994. A tour of the NASA LeRC 2.2-Second Drop Tower and SEL was conducted.

During the month of July a CCD array camera was used to image 10 thin filaments to determine the 2D temperature field in a flame, a method to be used in future drop tower tests. Images were obtained and the reduction of the data was begun. Software development is continuing in order to deduce the temperature and to apply small corrections for conductive and radiative losses along the filament. The survivability of the filaments was tested and a way to separate many filaments from the initial filament bundle was developed, using acetone and water solutions.

Dr. King from NASA Headquarters recommended that we interact with modellers in the NASA microgravity program, so we contacted Prof. Elghobashi from the University of California at Irvine, who agreed to provide

computations of unsteady strained flames with full chemistry and radiative losses. We will use his computations for comparison to our future data, to determine if the flamelet modeling approach is a valid approach. We also plan to work with Dr. K. Kailasanath, from NRL, who is doing direct numerical simulations of microgravity flames. We agreed to plan our joint programs to provide overlapping conditions, whenever possible. It appears that our experiment should be especially useful for the Large Eddy Simulation models, such as the one presented by Dr. Kailasanath at the Symposium. This model uses Direct Numerical Simulation to solve the exact Navier Stokes equations down to a certain grid scale, then applies a subgrid model to simulate the smallest scale phenomenon. We can provide measurements that are needed to develop the subgrid model, which is based on flame-vortex interactions.

During the month of August the research team met to plan the design of the drop tower experiment and arrange for the integration of the experiments and the model to be developed. A preliminary design of the experiment to be dropped was sketched out. Vendors were contacted to obtain information regarding the sizes of light sources, cameras, electronics and fuel tanks. Calculations were performed to determine the feasibility of certain diagnostics, including white light PIV, Mie imaging using oil drops, and thin filament pyrometry. Efforts also are being made to improve and apply the software needed for the PIV and Mie imaging diagnostics. Software development is progressing while the experiment is being designed and fabricated.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 5/94 EXPIRATION: 4/98  
PROJECT IDENTIFICATION: 962-22-05-45  
RESPONSIBLE CENTER: LeRC

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*Aerodynamic, Unsteady, Kinetic, and Heat Loss Effects on the Dynamics and Structure of Weakly-Burning Flames in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Fokion N. Egolfopoulos

University of Southern California

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

To obtain quantitative information (flame speed, velocity field, temperature) about the characteristics of weakly burning flames in microgravity, and to apply this information to analyze near-limit phenomena at normal gravity conditions.

**TASK DESCRIPTION:**

The experiments will employ a counter-flow burner geometry to provide a nearly adiabatic flame with a well-controlled strain, for conditions that cannot be achieved in normal gravity. Both premixed and diffusion flames will be studied at various pressures, and Laser Doppler Velocimetry, Particle Image Velocimetry and high-speed cinematography will be used to measure the flow fields. Models will be developed for these cases for comparison purposes. In addition, flame interactions will be studied, and numerical simulations of flame propagation in tubes in spherical vessels will be performed. Experiments will be conducted at normal gravity, in the NASA LeRC 2.2-Second Drop Tower, and aboard the NASA aircraft.

**TASK SIGNIFICANCE:**

The research will allow a first-time study of flames burning near their flammability limit (the concentration of fuel in air that just forms a flammable mixture) without the complications of buoyancy, wall heat losses, or time-varying strain. It will provide fundamental data about the flammability limits that can be used to verify theoretical calculations, and has applications to turbulent combustion such as that found in engines.

**PROGRESS DURING FY 1994:**

This is a new project, so effort has thus far been concentrated on defining the order of the experiments and designing hardware. Design and drawings for the counterflow burner apparatus have been completed, and construction is underway. Layout of the hardware in a drop frame for the 2.2-Second Drop Tower, testing of a new flame ignition system, and design of a PIV test rig are underway.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 5/94 EXPIRATION: 4/98

PROJECT IDENTIFICATION: 962-22-05-50

RESPONSIBLE CENTER: LeRC

### *Effects of Gravity on Sheared and Nonsheared Turbulent Nonpremixed Flames*

PRINCIPAL INVESTIGATOR: Prof. Said E. Elghobashi

University of California, Irvine

CO-INVESTIGATORS:

Dr. Y.Y. Lee

University of California, Irvine

Mr. R. Zhong

University of California, Irvine

#### TASK OBJECTIVE:

The objective is to answer fundamental questions regarding the effect of buoyancy on turbulent combustion. These questions include the following:

1. How does buoyancy affect the small-scale structure of the scalar and velocity fields?
2. How does buoyancy affect the distribution of scalar dissipation near the reaction zone?
3. Under what conditions is the flamelet model valid for buoyant turbulent diffusion flames?
4. How does flame extinction depend on the appropriate non-dimensional numbers ( $Fr$ ,  $Da$ ,  $Re$ , etc.), the magnitude of the reaction energy, and the direction of gravity relative the fuel-air interface?

#### TASK DESCRIPTION:

This research is entirely computational. It will focus on direct numerical simulation (DNS) of two cases of flow: in initially isotropic turbulent flow (without shear) and a flow with initially uniform shear. In both cases, separate, parallel streams of fuel and oxidizer will enter the computational domain and react. Numerical models that have already been developed by the Principal Investigator will be extended to these cases with principal additions to include finite rate chemistry and non-zero gravity. The results will be condensed in the form of diagrams based on the appropriate nondimensional numbers.

#### TASK SIGNIFICANCE:

This work will answer fundamental questions regarding how buoyancy induced by gravity affects turbulent combustion. In particular, gas, temperature, and velocity distributions can be very different in the absence of gravity, and this research will focus on revealing the small-scale structural differences in the flame, as well as probing the dependence of flame extinction on the alignment between gravity and the fuel-air interface. With this information, mathematical models of turbulent diffusion flames can be modified to better account for buoyancy effects. Better understanding of the physical processes will lead to improved combustor designs and enhanced predictive capabilities regarding fires on Earth and in a space environment.

#### PROGRESS DURING FY 1994:

This is a new research effort. Thus far, the partial derivative terms in the existing computer code have been modified from 2nd order central differences to 5th order and 6th order accurate schemes and the results checked against the exact solution of the Taylor-Green vortex flow. The conclusion was that while the new approach yielded more accurate results, the increase in computer time was not acceptable. Current efforts are under way to rewrite the code so that it can take advantage of new parallel processor computers, and to continue code additions to include finite rate chemistry and buoyancy forces.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 5/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-59

RESPONSIBLE CENTER: LeRC

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### *Combustion of Electrostatic Sprays of Liquid Fuels in Laminar and Turbulent Regimes*

PRINCIPAL INVESTIGATOR: Prof. Alessandro Gomez

Yale University

#### CO-INVESTIGATORS:

Prof. M.B. Long

Yale University

Prof. M.D. Smooke

Yale University

#### TASK OBJECTIVE:

This research is a combination of experimental and computational work by Professors Alessandro Gomez (PI, experimental), Marshall B. Long (Co-I, diagnostics) and Mitchell D. Smooke (Co-I, computational), all from Yale University. It involves studying the formation and burning of electrosprays of liquid fuels at both normal and reduced gravity.

#### TASK DESCRIPTION:

Normal-gravity testing of electrostatic sprays is conducted with cold-flows (i.e., non-buoyant) and in counterflow diffusion flames. Microgravity tests of the counterflow diffusion flame are conducted in the NASA LeRC 2.2-Second Drop Tower. A numerical model of a gas-phase counterflow diffusion flame is being extended to account for the droplet spray, for comparison of predictions with experimental results.

#### TASK SIGNIFICANCE:

The goal of this investigation is to study the combustion of well-defined and well-controlled sprays in configurations of successively increasing levels of complexity, starting from laminar sprays to fully turbulent ones. This may lead to improvements in the design of diesel engines or liquid-fueled rocket engines.

#### PROGRESS DURING FY 1994:

The FY94 research effort was focused on three areas: (1) tests with electrostatic sprays, (2) tests with sprays produced by an ultrasonic nebulizer, and (3) the development of laser diagnostics for droplet size measurements. There were no microgravity tests conducted in FY94 due to the shut-down of the NASA LeRC 2.2-Second Drop Tower.

##### (1) Experiments with Electrosprays

Normal-gravity tests were conducted with an electrostatic spray of heptane in an axisymmetric, laminar, diffusion flame. The flames were like a candle flame in appearance (i.e., sheath combustion), with a dark core (where the droplets evaporate without burning), surrounded by a high-temperature soot-shell, enveloped by the blue flame. There was no evidence of individual droplet combustion. The results (based on phase Doppler anemometry and sizing, as well as thermocouple measurements) indicate that the D-square law with constant evaporation rate was satisfied over a significant portion of the droplet history. It was found that the average evaporation rate could be determined from the height of the dark core without intrusive measurement. It was also observed that droplet-droplet interactions, although present near the flame surface, were apparently not significant in the flame core.

##### (2) Experiments with Sprays Produced by an Ultrasonic Nebulizer

Preliminary normal-gravity tests were conducted with an ultrasonic spray, since the electrostatic spray does not allow studies of flame extinction and droplet interaction under low-slip conditions (which are more amenable to numerical modeling). Preliminary tests indicate that the mean droplet diameter is independent of flow rate and controlled by the resonant frequency of the piezoelectric crystal. Good size distributions have been attained (with a relative standard deviation less than 0.3), although the droplets are not monodisperse as with the electrospray. A self-sustained flat flame has been established over a much wider range of conditions than possible with the electrostatic spray (e.g., 2 cc/min and a strain rate of 30 s<sup>-1</sup>), and work is underway to improve the burner stability. The researchers are now considering how to adapt the ultrasonic burner to the 2.2-second drop package.

(3) Development of Laser Diagnostics

Improvements have also been made in the measurement of droplet size. By matching the angular scattering pattern with calculations for spheres from Mie-Lorentz theory, size determination has been improved from an accuracy of 4% to 1% in droplet diameter (12% to 3% in volume). Since the comparison of the measurements with Mie-theory calculations is very time-consuming, algorithms were developed based on FFT to generate a look-up table associating scattering patterns with measurements. The sensitivity of the technique to the effects of finite laser beam size will be investigate in the future. A paper describing the technique is being prepared for submission to Applied Optics.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 3/91 EXPIRATION: 12/98

PROJECT IDENTIFICATION: 962-22-05-34

RESPONSIBLE CENTER: LeRC

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*Three-Dimensional Flow in a Microgravity Diffusion Flame*

PRINCIPAL INVESTIGATOR: Prof. Jean R. Hertzberg

University of Colorado, Boulder

## CO-INVESTIGATORS:

Mark A. Linne

Colorado School of Mines

**TASK OBJECTIVE:**

The objective is to study the influence of buoyancy on the three-dimensional fluid dynamics of the interaction between a diffusion flame and the elliptic vortex rings formed by pulsations of the fuel flow issuing from an elliptic nozzle.

**TASK DESCRIPTION:**

A methane diffusion flame burning in atmospheric conditions is investigated. Elliptic vortex rings are generated by a combination of periodic flow perturbations (active forcing) and the elliptic geometry of the fuel nozzle (passive forcing). The fuel flow is forced by an in-line 100 watt loudspeaker system at frequencies up to 1 KHz. Phase-locked flow visualization and particle image velocimetry (PIV) are the primary diagnostics.

**TASK SIGNIFICANCE:**

Techniques to control turbulent structures in flames are investigated with the objectives in mind such as enhancing combustion efficiency and reducing pollutant formation using techniques of active and passive control of fluid dynamics. Potential applications include industrial and residential burners and gas/liquid fueled engines.

**PROGRESS DURING FY 1994:**

Preliminary test plans and schedule were prepared and presented to LeRC's drop tower and diagnostics personnel.

Normal gravity studies on the prototype combustor revealed that the flow exhibits a bifurcation type phenomenon in the absence of flame. A nonreacting flow of air was visualized using cigarette smoke and alumina particles, illuminated by a strobe, phase-locked to the forcing signal. The results show that the acoustically forced jet forms a series of vortex rings which evolve into a complicated structure, which then splits into two or three jets several diameters downstream of the nozzle exit. Preliminary results suggest that this phenomenon exists over a wider range of flow rates and at lower forcing levels than the bifurcated flame phenomenon. The effective frequency range for the nonreacting air flow is also wider and lower than the flame phenomenon. The presence of the bifurcating phenomenon in a nonreacting air flow suggests that the driving mechanism is not an absolute instability, but is more likely a combination of axial and azimuthal forcing.

Efforts have begun to visualize the fuel jet in the bifurcated flame case. Note that techniques developed for the bifurcated flame will be applied to the asymmetric forced flame as appropriate. The fuel flow was seeded with aluminum oxide as in the previous non-reacting air flow, using a cyclone/fluidized bed seeder. A white-light strobe was phase-locked to the forcing signal. However, the flame emission was several times more intense than the light scattered by the seed, so further development of this technique is needed. As a first step, the experiment rig was moved to a different room, where a 12 watt argon ion laser is available. This laser will be chopped with a chopper wheel and attempts will be made to phase-lock it to the forcing signal. If this is unsuccessful, a Bragg cell based chopping system can be developed. Eventually, a diode laser system is planned in place of the (large) ion laser.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 6

MS Students: 0

PhD Students: 1

TASK INITIATION: 5/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-58

RESPONSIBLE CENTER: LeRC

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*Chemical Inhibitor Effects on Diffusion Flames in Microgravity*

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**PRINCIPAL INVESTIGATOR:** Prof. Simone Hochgreb

Massachusetts Institute of Technology (MIT)

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**CO-INVESTIGATORS:**

Gregory Linteris

National Institute of Standards and Technology (NIST)

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**TASK OBJECTIVE:**

The objectives of the proposed research are as follows:

1. To determine the effects of flame inhibitors on the physical characteristics (height, shape, color, and luminosity) and stability limits (ignition, extinction, lift-off and blow-off) of gaseous diffusion flames in the presence of halogenated fire suppressants in microgravity.

2. To develop quantitative analytical models for the observed behavior, including chemical kinetic effects, in order to understand the mechanisms of inhibition of halogenated compounds in gaseous diffusion flames.

**TASK DESCRIPTION:**

The experiments will consist of normal and microgravity studies of laminar jet and co-flow diffusion flames inhibited by halogenated suppressant addition to the air or fuel stream. In microgravity, visual and temperature diagnostics will be utilized to detect flame shape, dynamics and stability limits. Normal gravity experiments will make use of additional chromatographic and spectroscopic diagnostics in addition to visual and temperature experiments.

Analytical and computational work will be performed using existing general 2-D codes with chemical kinetic models for comparison with the observed results. The calculations should serve for comparison with observations, explanation of observations and selection of additional experimental conditions of interest.

**TASK SIGNIFICANCE:**

Fire suppression in space is one of the main reasons to investigate combustion phenomena in microgravity. Since halogenated compounds will remain as the fire suppressant of choice in many space missions, there is clearly a need for experimental evidence on the effectiveness of these compounds under zero buoyancy conditions.

The final product of the experimental and analytical work is a comprehensive and quantitative understanding of the physical and chemical phenomena involved in the suppression of diffusion flames through the addition of chemical halogenated inhibitors. This understanding is expected to be very useful both from a fundamental viewpoint as well as for the practical utilization of chemical fire suppressants in space.

**PROGRESS DURING FY 1994:**

In order to first examine the effects of the inhibitors CF<sub>3</sub>H and CF<sub>3</sub>Br on diffusion flames in normal gravity we constructed a co-flow diffusion flame burner capable of operation with variable jet diameters. Calibrated mass flow controllers under computer control metered the flows of air, fuel gas (propane and methane), and inhibitor. Two jet diameters were used in the current experiments (0.1 mm and 1.2 mm). The smaller jet produces a flame 0.3 to 0.7 cm tall, and the larger jet 14 to 16 cm. Because of the flame's small size, the effect of buoyancy will be less using the smaller jet, allowing some early assessment of the effects of buoyancy on the flame characteristics.

In these tests, we first determined the flow rate of methane necessary to cause blow-off for each jet diameter, and determined the effect of the air co-flow velocity on the blow-off condition. It appeared that the blow-off condition was sensitive to the aerodynamic conditions near the jet tip which were affected by the co-flow velocity and any



geometrically induced flow disturbances. We then measured the concentration of  $\text{CF}_3\text{H}$  and  $\text{CF}_3\text{Br}$  which caused blow-off at a methane flow rate of 50% of the fuel blow-off condition for these two jet diameters. The results of the experiments will help us to determine the initial test conditions of the NASA LeRC 2-Second Drop Tower tests in the presence of inhibitor. We next recorded video images of the flames as a function of either the fuel flow rate or the inhibitor concentration in the air stream, and used these to determine the flame height. These data can be used to assess the utility of simple analytical models for predicting the flame size.

An important component of the present project will be calculations of the flame structure using a detailed chemical kinetic mechanism and a suitable flame model. This month we acquired a kinetic mechanism for fluorine inhibition of hydrocarbon flames from Burgess/Tsang/Zachariah/Westmoreland at the National Institute of Standards and Technology (NIST) and combined it with GRIMECH obtained from Greg Smith at SRI. Using the premixed flame code of SANDIA, we have performed extensive tests of its performance against premixed flame speed measurements recently obtained at NIST and MIT.

1) Measurements of the effect of  $\text{CF}_3\text{H}$  and  $\text{C}_2\text{F}_6$  on  $\text{CH}_4$  laminar flame speeds in a combustion bomb at variable pressure and temperature have been concluded. The experimental data, at equivalence ratios of 0.8 to 1.2, pressures between 0.7 and 8 atm and temperatures between 300 and 500 °K show that, as shown for experiments at ambient conditions, both inhibitors are more effective at rich conditions,  $\text{C}_2\text{F}_6$  being twice as effective as  $\text{CF}_3\text{Br}$ . No particular dependence of the inhibition effect on temperature or pressure is apparent, the effects being primarily a function of inhibitor concentration (tested between 0 and 5 percent) and equivalence ratio. Results are currently being prepared for publication.

2) The chemical kinetic mechanism developed at NIST for  $\text{CF}_3\text{H}$  and  $\text{C}_2\text{F}_6$  inhibition effects was used to predict flame speeds at the varying pressure and temperature conditions for which the tests were conducted. The results show remarkable agreement under lean conditions, but predictions are 10-15 percent lower than observed values at the richest equivalence ratio. This leads us to conclude that some difficulties are potentially to be expected in modeling diffusion flames, where much of the chemistry takes place under rich fuel conditions.

3) Before we are able to use the NIST fluorinated species inhibition mechanism to model our inhibited methane- and CO-air diffusion flames, we must first test the accuracy of the mechanism. Towards this end, we have begun testing the mechanism against both data collected at NIST and with data existing in the literature. This month we performed extensive calculations of the flame structure of premixed methane-air and CO-O<sub>2</sub>-Ar flames inhibited by  $\text{CF}_3\text{H}$ . The methane calculations were performed at  $\phi=0.9, 1.0$ , and  $1.1$  for inhibitor mole fractions up to 8%, and these were compared with recent measurements at NIST. The NIST mechanism was found to predict several of the important features of the  $\text{CF}_3\text{H}$  inhibition, including the strong effect of equivalence ratio on the burning rate reduction (rich flames are inhibited more), and a reduction in inhibitor effectiveness at higher inhibitor mole fractions. The kinetic mechanism, however, is predicting about 30% more reduction in burning rate than the experiments show. We have been working closely with Wing Tsang and Don Burgess to try to determine the possible areas for improvement in the mechanism.

4) In order to test the effectiveness of  $\text{CF}_3\text{Br}$  and  $\text{CF}_3\text{H}$  in micro-gravity under non-sooting conditions, we plan to study inhibited CO flames. The burning rate reduction of premixed CO-O<sub>2</sub>-Ar flames by  $\text{CF}_3\text{H}$  has been measured by Vandooren and co-workers. To test the model for moist CO oxidation, we have performed numerical calculations of the burning rate for all of Vandooren's conditions (about 150 flames). In nearly all of these calculations, the burning rate was predicted within 10% (flames with very low H<sub>2</sub> concentration were not well predicted). Hence, it appears that the model works very well for moist CO flames, but may need more work for CH<sub>4</sub> flames.

5) We have started to set up a counter-flow diffusion flame for studies of  $\text{CF}_3\text{H}$  and  $\text{CF}_3\text{Br}$  inhibited diffusion flames, again, to test the mechanism, and to start to examine the mechanism to study the effects of transport on the inhibition.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 2

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TASK INITIATION: 6/94 EXPIRATION: 6/98

PROJECT IDENTIFICATION: 962-22-05-54

RESPONSIBLE CENTER: LeRC

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*Time-Dependent Computational Studies of Pre-Mixed Flames*

PRINCIPAL INVESTIGATOR: Dr. K. Kailasanath

Naval Research Laboratory (NRL)

CO-INVESTIGATORS:

Dr. G. Patnaik

Berkeley Research Associates

Dr. E.S. Oran

Naval Research Labs

**TASK OBJECTIVE:**

Study the structure (multidimensional) and dynamics (propagation, stability and extinction) of premixed flames in microgravity and normal gravity environments.

**TASK DESCRIPTION:**

Perform detailed numerical simulations using time-dependent, one-dimensional and two-dimensional flame models. These models solve the multispecies-coupled, partial-differential, reactive-flow equations, including fluid, thermodynamic and transport properties, chemical kinetics, radiative heat transfer, buoyancy and boundary heat loss effects. The investigator will a) systematically isolate and evaluate the relative importance of various physical and chemical processes that may be controlling the structure and dynamics of premixed flames, and b) compare qualitatively and quantitatively with experimental observations.<sup>1</sup>

**TASK SIGNIFICANCE:**

Small amounts of fuel leaking from fuel cells could pose a fire hazard on the space-shuttle and similar vehicles. Therefore, understanding the burning and extinguishment characteristics of flames are important for the safe operation of space vehicles. The knowledge gained from such studies is also useful for the safe and efficient use of fuels on Earth. Computer simulations are a cost effective way to obtain data to corroborate limited experimental observations and provide additional details which are difficult to obtain from space based experiments.

**PROGRESS DURING FY 1994:**

Investigations of burner-stabilized lean methane-air flames indicate that a 5% methane-air flame cannot be stabilized even by including the effects of radiative losses. This is in contrast to freely-propagating flames which can be sustained in this mixture, with and without the inclusion of radiative losses. However, a 5.5% methane-air flame can be stabilized on a burner, but only for high inflow velocities such as 7 cm/s, which is close to the freely-propagating burning velocity. When the inflow velocity is reduced to 6 cm/s, the flame shows a longitudinal oscillation with excursions of the flame temperature above and below the planar adiabatic values and eventually dies. These oscillations are independent of the spatial and temporal resolution of the numerical solution. They appear to be a manifestation of the transition from the imposed initial condition of a propagating flame to its stable no flame final condition. However, these oscillations do not seem to be very significant physically since the flames eventually die. For lower inflow velocities, the flames extinguish more quickly. Overall, the simulations suggest that conductive and radiative heat losses play an important role in the stability of very lean methane-air flames and that such flames can be stabilized on burners only for a narrow range of inflow velocities.

Some of the characteristics of freely-propagating low velocity lean methane-air flames are quite distinct from those of lean hydrogen-air flames with similar velocities. The methane flames are thinner and have significantly higher flame temperatures.

The cellular structures observed in lean methane-air mixtures (5-6%) were very weak when compared to those in lean hydrogen-air flames of comparable burning velocities. Therefore, after discussions with Prof. Paul Ronney, a CO<sub>2</sub> diluted methane-oxygen mixture, with an effective Lewis number of about 0.70, was chosen for studying a case which should exhibit strong cellular structures. Both the simulations and Ronney's experiments indeed indicate well defined cellular structures. However, the detailed structures are not quite the same in the two cases. Computed

burning velocities are larger than the experimental ones; however, the flame temperatures are about the same. Three-dimensional effects, insufficient numerical resolution and inadequacy of the chemical kinetics scheme to adequately capture the effects of CO<sub>2</sub> dilution are possible explanations for the observed differences.

The variation of the burning velocity with dilution is being studied to see if the "skeletal" mechanism used in the computations is adequate to represent the chemical effects of dilution with CO<sub>2</sub>. This work is hampered by the lack of experimental data on CO<sub>2</sub> dilution effects. Further tests with N<sub>2</sub> dilution are underway to check if the reaction scheme is adequate for any dilute mixture. Tests in which the equivalence ratio was varied showed that the scheme was adequate for those cases.

Three-dimensional simulations as well as simulations using more complex chemistry schemes will need parallel processing to be efficient. One of the major problems in achieving efficient parallel processing is distributing the work-load among the various processors. The first step towards the development of a flame code that will efficiently run on massively parallel processors such as the Intel/IPSC 860 was to develop a load-balancing procedure for the chemistry. This has been accomplished using the newly developed Work-Parti software from the University of Maryland. 'Stiff' points are gathered into a 1-D array, and only those points are redistributed to other processors using gather/scatter operations. This tends to reduce the number of points needed to be moved to achieve the load balance. After tackling the chemistry, the FCT fluid convection, radiation, inflow velocity control and timestep control routines were rewritten to make it more efficient and reduce the scratch space. David Fyfe (NRL) and Craig Douglas (IBM) were instrumental in re-writing the multigrid part of the code for parallel implementation. Then, the diffusive transport modules were also parallelized.

The development and testing of a fully parallel version of the flame code has been completed successfully. The code has been run on the Intel/IPSC 860 at NRL and the Intel Paragon at Air Force Wright Aeronautical Labs and shown to produce the same results as on the Cray C-90.

The newly developed parallel version of the flame code is being used to study the detailed dynamics of the extinguishment of downward-propagating methane-air flames. The plan is to compare these observations to those made in the earlier NRL study of the extinguishment of hydrogen-air flames. Such a comparison will be helpful in isolating effects that are specific to the highly diffusive and easily combustible hydrogen-air mixtures from those that are more general and applicable to a variety of fuel-air mixtures. Furthermore, it will also provide some information on the relative role of conductive and radiative heat losses in extinguishing flames propagating in tubes. The earlier NRL studies of one-dimensional, zero-gravity flames in lean methane-air mixtures showed that radiative losses alone can extinguish some flames. In this study of two-dimensional flames, radiative and conductive losses will be considered together and separately to isolate their effects. The NRL simulations to date show that a 5% methane-air flame propagates without extinguishment even when considering both losses, so leaner mixtures will be tried next.

Currently, studies of near limit downward-propagating methane flames are continuing. Discussions with Prof. Paul Ronney and others at the International Combustion Symposium identified the third-body efficiencies in the chemistry scheme as a possible source of the differences observed earlier between Paul's results and ours for the extinguishment of CO<sub>2</sub> diluted methane flames. It was decided to pursue this matter further using the FLAME1D code before performing more detailed multidimensional simulations of the extinguishment of methane-air flames. Different values for the third body efficiencies suggested in the literature have been incorporated into the codes. A repeat of stoichiometric methane-air flames shows that these parameters do not make any difference in the results. However, a repeat of the simulations of the CO<sub>2</sub> diluted mixtures used in Paul's experiments shows that these parameters have a strong influence on the flame temperatures and velocities. In fact, it has been difficult to even ignite some of these mixtures. Our results to date show that the burning velocities of near-limit CO<sub>2</sub> diluted mixtures are now lower than prof. Ronney's. Recall that with the efficiencies set to unity, our burning velocities were higher than those observed in his experiments. It appears that the exact values of the third body efficiencies could play a crucial role in obtaining accurate results. It is not clear how accurately these parameters are known. Preliminary calculations with a 5% methane-air mixture now show that it is very difficult to obtain a steadily propagating flae in this mixture when radiation and non-unity third-body efficiencies are taken into account. Further calculations are being performed to resolve this issue.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 2/92 EXPIRATION: 12/97

PROJECT IDENTIFICATION: 962-22-05-22

RESPONSIBLE CENTER: LeRC

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**Journals**

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Patnaik, G. and Kailasanath, K. Effect of gravity on multidimensional burner-stabilized flames, in Heat Transfer in Microgravity, eds. C.T. Avedisian and V.A. Arpaci. HTD. the American Society of Mechanical Engineers, New York, NY, vol. 269, 79 (1993).

**Presentations**

Patnaik, G. and Kailasanath, K. "Effect of gravity on multidimensional burner-stabilized flames." ASME Winter Annual Meeting, New York, NY, 1993.

Patnaik, G. and Kailasanath, K. "Cellular structure of lean hydrogen and methane flames." ASME Winter Annual Meeting, New York, NY, 1993.

Patnaik, G. and Kailasanath, K. "Numerical simulations of burner-stabilized hydrogen-air flames in microgravity." the 25th Int. Symp. on Combustion, Irvine, CA, July 31 - August 4, 1994.

Patnaik, G. and Kailasanath, K. "Cellular structure of lean hydrogen and methane flames." AIAA 94-3275, 30th AIAA/ASME/ASME/SAE Joint Propulsion Conference, Indianapolis, IN, June 27-30, 1994.

Patnaik, G. and Kailasanath, K. "Numerical simulations of burner-stabilized hydrogen-air flames in microgravity." presented at the 25th Int. Symp. on Combustion, Irvine, CA, July 31 - August 4, 1994.

### *Radiative Ignition and Transition to Flame Spread in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Takashi Kashiwagi

National Institute of Standards and Technology (NIST)

#### CO-INVESTIGATORS:

Dr. H. Baum  
S.L. Olson

National Institute of Standards and Technology (NIST)  
NASA Lewis Research Center (LeRC)

#### TASK OBJECTIVE:

The objectives of the Middeck Glovebox RITSI Investigation is to conduct an experimental study of the radiative ignition and subsequent transition to flame spread in low gravity in the presence of very low speed air flows in 2D and 3D configurations and to compare the experimental observations with the theoretical model.

Although the fundamental processes involved in radiative ignition have been suggested in the literature, there have been no definitive experimental or modeling studies up to this point due to the flow motion generated by buoyancy (starting plume problem) near the heated sample. In addition, there are very few studies of the transition from ignition to flame spread. Almost all flame spread studies are based on two-dimensional, quasi-steady or steady flame spread. The limit on the viability of flame spread should be controlled by the transient process from ignition to flame spread. Steady-state flame spread models cannot be expected to accurately describe this limit.

For the first time the use of a prolonged microgravity environment allows us time to conduct a definitive study on ignition and subsequent transition to flame spread. The model and numerical codes for such a calculation are nearing the end of their development, and experimental data is needed to compare with the predicted results. From the comparison, we will learn how accurately we understand ignition and flame spread mechanisms and where the deficiencies in our understanding of them might exist.

#### TASK DESCRIPTION:

RITSI hardware uses a localized irradiated area via a radiant heater and/or hot wire to ignite samples of cellulose of various thickness and/or composition. The hardware will provide a means to record ignition delay time and the subsequent transition to flame spread at various low speed air flows. It will also provide a means to record solid and gas-phase temperature data as well as the radiant heater and flow information.

Variables in priority order are 1) Flow velocity (0,2,5 cm/s); 2) Irradiated sample diameter (1 cm, 2.5 cm) and/or power level (up to 8 W/cm<sup>2</sup>) 3) Smoldering via metal ion doping of fuel 4) Geometry of sample (2D or 3D) and 5) Sample thickness.

RITSI hardware consists of a flow duct with screens at both ends and a fan (pulling air through duct rather than pushing it) at one end along with an area reducer to provide the air flow. One side of the duct opens for access to the sample holder for changeout of samples in the glovebox. Some samples are narrow rectangles, for upstream and downstream propagation only, and some will be more square, for propagation sideways as well.

#### TASK SIGNIFICANCE:

In previous studies, ignition and flame spread were studied separately with the result that there has been little understanding of the transition from ignition to flame spread. In spacecraft fire safety applications this transition is crucial to determine whether a fire will be limited to a localized, temporary burn or will transition into a growth mode with the potential to become a large fire.

### PROGRESS DURING FY 1994:

(October 1993) RITSI: Formal approval from NASA HQ based on the September nonadvocate peer review was received for continued development of a Glovebox experiment called RITSI (Radiative Ignition and Transition to Spread Investigation). The engineering unit assembly is beginning electrical assembly.

(November 1993) Theoretical study: the variable mesh code is working and quiescent three-dimensional calculations are being run. RITSI: The engineering unit assembly is complete and calibrations are underway in preparation for December Learjet low gravity testing.

(December 1993) RITSI: Six Learjet flights with 14 tests were successfully conducted. Ignition of plain Whatman 44 was not achieved with the lamp alone, and modifications to the ignition procedure allowed for successful ignition with a soot spot and gas-phase pilot in addition to the lamp. Smolder-promoted paper ignited easily.

(January 1994) RITSI: Data analysis of the Learjet tests by LeRC personnel is underway in preparation for a visit to NIST by LeRC's S. Olson.

(February 1994) Theoretical: Axisymmetric cases were run with a variable step size, and the results documented in a Combustion and Flame paper (1994). RITSI: Preliminary results of the Learjet tests were presented to NIST during a February 17 visit by S. Olson. Flight hardware design has started.

(March 1994) Formal approval of a NRA-submitted proposal was received by the PI from NASA HQ. A flight experiment as a follow-on to the precursor RITSI Glovebox experiment is approved. This experiment is tentatively entitled TIFS-3D (Transition from Ignition to Flame Spread in 3D). The Lewis team (Project Scientist and Project Engineer) were assigned. Theoretical: A pilot ignitor in the gas-phase was added to the code. Variations of the incoming velocity distribution are being studied. RITSI: Odor testing was conducted. Data analysis and upgrades of the engineering hardware continue.

(April 1994) Theoretical: Numerical computations have been run to evaluate the effect of energy distribution on the ignition and transition to flame spreading quiescent environments. Spot size and total energy were varied, and the results indicate that although the final flame spread rate is independent of energy distribution, the ignition delay and the transient spread rate from ignition to steady state are strong functions of the energy distribution. RITSI: Flight hardware design is nearly complete. Engineering testing of ignition delay and ignitor wire temperature was conducted. Negotiations for testing of the Engineering Unit at Brunel to determine production levels of CO and the Glovebox facility's ability to convert the CO to CO<sub>2</sub> were initiated. TIFS-3D: Project team reviewed proposal. Negotiations with Japan were initiated on a collaboration to conduct tests with the RITSI Engineering hardware in the 10 second Hokkaido JAMIC facility.

(May 1994) Theoretical: 2D flame ignition and subsequent flame spread of a finite-width (uninhibited edges) thermally-thin paper were calculated with no wind. The results showed that the flame spread rate at the edge is at least twice as fast as the spread rate in the center of the paper. RITSI: Toxicity testing of the smolder-promoted paper for CO is being prepared at NIST. The output of the radiant heat lamp was experimentally characterized. TIFS-3D: Olson attended the kickoff meeting for the new flight NRA's. An Objective and Scope for TIFS-3D were drafted.

(June 1994) RITSI: Electrical and mechanical upgrades to the engineering hardware were complete. Verification documentation for the flight experiment was drafted. Additional lamp characterization tests were conducted. Status was reviewed with the PI during a visit by the PI on June 3. TIFS-3D: A memo was sent to HQ by the PI and Project Scientist requesting an expanded scope for the TIFS-3D experiment to include O<sub>2</sub> and preheating effects. The PI visited the project team on June 3 to define the experiment objectives, scope, draft matrix, and diagnostics requirements.

(July 1994) RITSI: Toxicity testing was completed at NIST. The negotiations for toxicity and abatement testing at Brunel are continuing. A manuscript of wind effects on flame spread is being prepared (joint theoretical and experimental). TIFS-3D: The project obtained 2 LeRC civil servants to work on preliminary breadboard flow and laser heater systems.

(August 1994) Theoretical study: A paper "Effects of Ignition and Wind on the Transition to Flame Spread in a Microgravity Environment" by K.B. McGratton, T. Kashiwagi, H.R. Baum, and S.L. Olson is completed and it is under internal review prior to submission to Combustion and Flame. RITSI: More smolder-promoted papers were made and sent to NASA LeRC for Learjet testing, normal gravity ignition testing, and CO toxicity testing at Brunel University as part of the flight hardware verification testing. TIFS-3D: The project team met to discuss design options.

(September 1994) Theoretical study: The internal review of the above paper has been completed after some modifications corresponding to comments by the reviewers. An extended abstract based on the paper has been written and submitted for presentation in the Eastern States Combustion Institute Meeting. RITSI: DC-9 Aircraft tests were conducted where both plain and smolder-promoted paper samples were successfully ignited in low gravity. The PI attended the First Meeting of NASA/NEDO Microgravity Combustion Coordinating Group for possible microgravity experiments using the Japanese 10 seconds drop tower. Agreement was reached that this was a fruitful endeavor, and a formal brief proposal will be submitted to the MCCG for formal approval of this venture. The PI also reviewed the flight hardware design progress and the engineering hardware test results. TIFS-3D: We had an informal meeting at NASA Lewis Research Center for discussing possible experimental parameters and diagnostic measurements. We also discussed an outline of SRD for this experiment. A CO<sub>2</sub> laser, a candidate radiant heater, was successfully used to ignite paper with no soot spot or gas-phase hot wire pilot

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 4/91 EXPIRATION: 12/97

PROJECT IDENTIFICATION: 962-22-05-35

RESPONSIBLE CENTER: LeRC

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*Sooting Turbulent Jet Diffusion Flames*

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PRINCIPAL INVESTIGATOR: Prof. Jerry C. Ku

Wayne State University

CO-INVESTIGATORS:

P.S. Greenberg

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objectives of this study are to model soot formation and radiation for turbulent jet diffusion flames and to determine the modeling coefficients from measured data under both normal and reduced-gravity conditions.

**TASK DESCRIPTION:**

In regard to experimental measurements, thermophoretic particle sampling and electron microscopy are used for soot particle size and aggregate morphology analysis. Laser light absorption imaging provides for the determination of soot-volume fractions, and emission imaging and thermocouple measurements will be used for soot thermometry. Laser Doppler Velocimetry may possibly be employed to measure velocities and turbulence intensities, but is beyond the stated deliverables of this effort.

In the area of modeling, Favre-averaged boundary layer equations with a k-e-g turbulence model and the conserved scalar approach with an assumed pdf (probability density function) are used to predict flow field and gaseous species mole fraction profiles, respectively. Transport of soot particles is described by equations for volume fraction and number density using rate equation models. The energy equation is included to provide coupling between flame structure and radiation analyses. The radiative flux is solved from the radiative transfer equation (RTE).

**TASK SIGNIFICANCE:**

Microgravity combustion is not only relevant to fire safety on-board a spacecraft but also provides a unique condition for better understanding of fundamentals of this common type of combustion.

**PROGRESS DURING FY 1994:**

The YIX method was applied to solve radiative heat transfer in a finite cylindrical enclosure with a nonhomogeneous, nongray, emitting, and absorbing soot/CO<sub>2</sub> mixture. Numerical results were obtained for radiative heat transfer within a turbulent ethylene jet diffusion flame from precalculated flame temperature, soot volume fraction, and CO<sub>2</sub> concentration maps. Soot only, CO<sub>2</sub> only, and combined cases were examined over the spectral range of 1-20 microns. Soot absorption coefficient spectra were calculated from soot volume fraction and refractive indices according to the Drude-Lorentz dispersion model based on the three frequently cited dispersion parameter sets. Results from these three dispersion parameter sets show that the difference in maximum flux is 51%. Thus, current uncertainties about soot spectral refractive indices are the main limitation on accurate estimates of the radiation heat transfer from sooting combustion systems. The exponential-wide-band model is used to calculate the CO<sub>2</sub> absorption coefficient spectrum. Overall, the contribution of soot radiation is only two to three times that of CO<sub>2</sub>. Consequently, both soot and CO<sub>2</sub> contributions are significant, and must be accounted for. For CO<sub>2</sub> gas, only contributions around absorption bands at 2.7 and 4.3 microns are significant. It seems that spectral contributions from the range above 5 microns may be neglected without any significant loss of accuracy in evaluating total radiation properties. This work was submitted to the ASME Journal of Heat Transfer. In addition, an improved method for handling the discontinuity between the velocity of fuel and co-flow has been implemented. The agreement between model prediction and Prof. Santoro's co-flow soot volume fraction data (already in the literature) is very good now.

The fabrication of a coaxial, piloted burner for turbulent gas jet diffusion flame studies has been completed. Extremely small tubing diameters (n 0.75 mm inner diameter for the inner jet) were employed to obtain large

Reynolds numbers while retaining a reasonable overall flame height compatible with the dimensions of the combustion chamber on the present 2.2 second laser rig. The major challenge was to provide a stable, coaxial arrangement of the inner jet and pilot nozzles. This was accomplished by the staff of the LeRC instrumentation shop, who employed an arrangement of laser-welded thin wire spacers to accurately control the relative position of the inner and outer tubes. After welding the spacers in place, the thin wires were then ground to the appropriate diameter to fit accurately into the pilot tube. The spacers also serve as flow straighteners for the pilot flow. Initial tests indicate that adequate symmetry has indeed been achieved.

A series of drops were conducted in the NASA LeRC 2.2 -Second facility to obtain the soot volume fraction in reduced gravity laminar jet diffusion flames. Tests indicated a reduction in local volume fraction for 1.5 mm jets using ethylene as a fuel at flow rates between 1.0 and 3.0 cc/sec. This resulted in volume fraction signals (obtained via light extinction measurements) with poor SNR's. Larger diameter burners were fabricated to provide longer optical path lengths, thus increasing signal strengths. Limitations on flow rate due to the capacity of the rig and the resulting flame lengths proved to be disadvantageous because stable flames could not be achieved in the available test time. Apparently Reynolds number considerations separate two different regimes wherein these flames are either diffusion or momentum dominated. The former evolve too slowly to be studied in the 2.2 second facility; the latter produce insufficient volume fractions for tractable flow rates and flame heights. Switching to acetylene as the fuel increased the soot production to the level wherein reasonable SNR's could be obtained. Some issues relating to the stability of these flames as a function of burner diameter and flow rate under reduced gravity conditions remain, and are presently being pursued. The hardware has been performing consistently well, as has the facility's newly refurbished air-bag deceleration system.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 2

TASK INITIATION: 3/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-22-05-36

RESPONSIBLE CENTER: LeRC

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*Soot and Radiation Measurements in Microgravity Turbulent Jet Diffusion Flames*

PRINCIPAL INVESTIGATOR: Prof. Jerry C. Ku

Wayne State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this study are to determine modeling coefficients from measurements of soot morphology and radiation in both normal and reduced-gravity turbulent gas jet diffusion flames, and to further refine models for soot formation and spectrally dependent radiation properties.

**TASK DESCRIPTION:**

In the area of experimental measurements, thermophoretic particle sampling and subsequent transmission electron microscopy analysis are used for soot particle size and analysis of aggregate morphology. Laser light absorption imaging provides for the determination of soot volume fractions. Emission imaging using isolated bandpass filters will be used to measure spectrally dependent soot radiation properties and possibly temperature. Rapid insertion of fine-wire thermocouples will also be used for the determination of temperature.

In the area of modeling, Favre-averaged boundary layer equations with a k-e-g turbulence model and conserved scalar approach with an assumed probability density functions (pdf) are used to predict flow field and gaseous species mole fraction profiles, respectively. The soot formation model has been modified and tested to predict soot volume fraction and number density. The energy equation is included to provide a full coupling between flame structure and radiation analysis. A third-order spherical harmonics approximation and the YIX method have been applied to solve the radiative transfer equation. In the proposed study, soot formation and radiation models will be improved, and methods for efficient spectral integrations and iteration between the solutions for the flame structure and the radiative transfer equation will be sought.

**TASK SIGNIFICANCE:**

Microgravity Combustion is not only relevant to fire safety on board a spacecraft, but also provides a unique condition for a better understanding of combustion fundamentals of this common type of combustion.

**PROGRESS DURING FY 1994:**

There is no progress to report at this time as the project has not started yet.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/95 EXPIRATION: 12/97

PROJECT IDENTIFICATION: 962-22-00

RESPONSIBLE CENTER: LeRC

*Studies of Flame Structure in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Chung K. Law

Princeton University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this work are to understand and quantify the structure, stabilization mechanisms, soot formation in, and extinction of one-dimensional premixed and nonpremixed laminar flames.

**TASK DESCRIPTION:**

This program comprises two main elements:

1. A numerical simulation of one-dimensional laminar flames is to be developed that, in addition to including the usual fluid mechanical and heat transfer mechanisms, will include detailed chemical kinetic mechanisms for comparison with the unique experimental results.
2. A drop-tower test apparatus is to be used to observe premixed laminar flames stabilized about cylindrical and spherical porous burners to distinguish heat loss and flow divergence influences on flame stabilization and flamefront stability.

**TASK SIGNIFICANCE:**

The one-dimensional adiabatic laminar unstretched premixed flame is a fundamental precept of combustion science, but cannot be stabilized in normal gravity because of the straining or asymmetrical influences of gravitationally induced buoyant convection. In microgravity experiments, this program demonstrates this combustion paradigm and provides a capability for probing the structure, chemistry, soot dynamics, and flame propagation speed of these fundamental flames.

**PROGRESS DURING FY 1994:**

This task was renewed in Fiscal Year 1994 with the award of an extended ground-based program entitled, "Studies of Flame Structure in Microgravity," under NRA-93-OLMSA-1, "Microgravity Combustion Science: Research and Flight Experiment Opportunities."

Progress on these two tasks are inseparable one from the other since the latter award is a simple continuation of the former, and together consist of:

1. A series of tests were completed providing observations of cylindrical, premixed flames in microgravity. These tests provided the first observations of flames stabilized near a burner by virtue of the flow divergence only and not by heat losses to the burner surface, as observed in normal gravity flame holding devices. This experimental work has provided a new method for evaluating the adiabatic flame speed for premixed flames, a fundamental property of fuels.
2. The formulation of an asymptotic theory of spinning premixed flames is completed. This work extends the previous diffusion flame analysis by Matalon (of a spinning fuel droplet undergoing diffusional burning in an oxidizing environment) to the premixed flame situation, wherein a mixture of fuel and oxidizer is ejected from a spherical source and subsequently burns as a premixed flame. This work is performed in anticipation of future experiments involving spinning premixed flames which are subjected to differing body forces and strain rates over the flame surface.

3. The theory for spinning spherical flames was investigated, including four cases that span: premixed and unpremixed fuels and fuels that require heat for vaporization (as in droplet combustion or monopropellant spheres) or those that do not (porous burner for gaseous fuels). This theory provides a basis for evaluating body force effects and flame stretch simultaneously. Preliminary results indicate a Lewis number dependence for the premixed case in which the flame flattens at the pole or at the equator; and a demonstration of extinction for unity Lewis number diffusion flames.

4. Experimentally, a new spherical burner has been developed using sintered metal, with flow incoming from both polar positions. Observed flame fronts in normal gravity are not smooth, which may be caused by nonuniform flow or by intrinsic flamefront instabilities.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/90 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 962-22-05-37

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

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Eng, J.A., Law, C.K. and Zhu, D.L. "On burner-stabilized cylindrical premixed flames in microgravity." Twenty-Fifth symposium (international) on Combustion, the Combustion Institute, presented, to appear, 1994.

*Structure and Dynamics of Diffusion Flames in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Moshe Matalon

Northwestern University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of the work are to gain insight into diffusion flames by studying simple combustion systems. The emphasis of the work will be to understand the structure and dynamics of stationary spherical flames, and the coupled processes occurring in the liquid and gas phases associated with the burning of liquid fuels.

For gaseous flames, the Principal Investigator will investigate the processes that lead to extinction of these flames either by the process of blowoff or by radiative losses. He will also examine the nature of the interactions between two diffusion flames, the mechanisms leading to the generation of flame front instabilities, the formation of cellular flames and the effect of residual gravitational acceleration on microgravity diffusion flames.

For liquid fuel burning, the Principal Investigator will examine the possible generation of instabilities that are intrinsic to the vaporization process and identify the importance of thermocapillary motion on the overall burning process.

**TASK DESCRIPTION:**

The program is entirely theoretical in nature. The Principal Investigator will work closely with related experimental investigations in the microgravity combustion science program to identify problems of specific interest.

The porous sphere will be the model problem for gaseous diffusion flames. A standard formulation of the conservation equations in spherical coordinates forms the base case. The model will then be extended to incorporate finite rate kinetics through activation energy asymptotics, enabling details of the flame structure to be elicited. The model can then be extended to include a standard form of a radiative loss to determine the extinction limits, blowoff and radiative, of the combustion system. Finally, the model will be extended to determine the effects of interaction by modeling two spherical diffusion flames in close proximity to each other employing bi-spherical coordinates in the approach outlined above.

As a next step the core model will be extended to incorporate stability theory and determine the conditions for the onset of flame-front instabilities similar to those that have been observed experimentally. Similarly, the effect of a low-magnitude, time-varying body force (g-jitter) on the flame will be examined, by casting the disturbance as a sinusoidal term in the governing equations.

The ideas developed above will then be extended to liquid fuels. The major change to the model will be the incorporation of the liquid phase, the conservation equations for the liquid phase, and the coupling of the gas and liquid phases. A major addition will be the incorporation of thermocapillary motion in the liquid phase equations.

**TASK SIGNIFICANCE:**

The proposed research will lead to a greater understanding of diffusion flames in general. By working closely with experimental studies in the microgravity science program, the investigator will be able to give a theoretical foundation to many experimental observations of diffusion flames in microgravity.

## PROGRESS DURING FY 1994:

Currently, the participants in this program are Prof. M. Matalon (the PI) and two Ph.D. students. The work which has been initiated is associated with 1) flame instability in pool burning, and 2) diffusional-thermal instabilities in diffusion flames. For the first problem, a simple one-dimensional model has been developed describing the evaporation and subsequent burning from a flat bed of a liquid fuel. The evolution of small disturbances is being examined in order to identify possible instabilities. Experiments show that while one possible mode of burning is a pool burning, in some circumstances a traveling front along the fuel bed may occur. The goal of the theoretical investigation is to explain these observations. The analysis of the second problem requires a complete understanding of the structure of a corrugated (three-dimensional) diffusion flame with arbitrary Lewis numbers (not necessarily = 1). Previous work, notably that of Linan, has been restricted to one-dimensional flame fronts and to unity Lewis numbers. It should be mentioned that while diffusional-thermal effects are reasonably well understood for premixed flames, they are not well understood for diffusion flames. We have thus extended Linan's analysis and gave a description for the structure of a diffusion flame of arbitrary 3D shape. Preliminary results indicate that in the absence of fuel or oxidant leakage through the reaction zone, the diffusion flame is absolutely stable. Instabilities are possible when leakage is present for a special range of the fuel and oxidant Lewis numbers and for a Damkohler numbers that is near the critical value corresponding to extinction. We are currently mapping the region of instabilities in terms of these parameters.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 6/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-47

RESPONSIBLE CENTER: LeRC

*Filtration Combustion for Microgravity Applications: (1) Smoldering, (2) Combustion Synthesis of Advanced Materials*

PRINCIPAL INVESTIGATOR: Prof. Bernard J. Matkowsky

Northwestern University

CO-INVESTIGATORS:

A. Bayliss

Northwestern University

V.A. Volpert

Northwestern University

**TASK OBJECTIVE:**

The objective is to investigate combustion in porous media with applications to (1) smoldering and (2) combustion synthesis of advanced materials, also referred to as self-propagating high-temperature synthesis (SHS).

**TASK DESCRIPTION:**

1. Proposing/developing theoretical models describing the fundamental mechanisms for the phenomena under consideration, (2) performing complementary analytical and numerical work on the proposed models and mechanisms, (3) comparing the results of these analyses to experiments.

**TASK SIGNIFICANCE:**

Both smoldering and combustion synthesis of advanced materials (SHS) contribute to fundamental science, and also represent important applications for microgravity combustion science; in smoldering to fire safety in both normal and microgravity, and in SHS to the determination of optimal synthesis conditions in both normal and microgravity environments. Combustion synthesis appears to compete favorably with conventional technology, by achieving shorter synthesis times, and at lower cost, by employing the internal energy of the combustion reactions rather than the costly external energy of a furnace, employed in conventional technology.

**PROGRESS DURING FY 1994:**

A paper entitled "Combustion of Porous Samples with Melting and Flow of Reactants," by A. Aldushin, B. Matkowsky, K. Shkadinsky, G. Shkadinskaya, and V. Volpert is accepted for publication by Combustion Science and Technology. The abstract of the paper reads: "We formulate and analyze a model describing the combustion of porous condensed materials in which a reactant melts and spreads through the pores of the sample. Thus there is liquid motion relative to the porous solid matrix. Our model describes the cases when the melt either fills all the pores or spreads through only some of them. In each case the melt occupies a prescribed volume fraction of the mixture. We employ both analytical and numerical methods to find uniformly propagating combustion waves, to analyze their stability and to determine behavior in the instability region. The principal physical conclusion which follows from our analysis is that the flow of the melted component can result in nonuniform composition of the product. Unlike models which do not take into account the relative motion of the components, this model exhibits a dependence of the structure of the product on the mode of propagation of the combustion front. Thus, if the initial mixture is uniform, models which do not allow for relative motion necessarily lead to uniform structure of the product, while in the model employed here the structure can be nonuniform. We observe that the structure of uniformly propagating combustion waves depends on whether the refractory or melting component is in excess in the initial mixture. We determine how various parameters of the system affect stability and find a pulsating instability of the uniformly propagating solutions. We also perform numerical simulations in order to (i) study the dynamical behavior of the combustion wave in the instability region, (ii) obtain a description of the melt flow on the scale of the entire sample rather than on the scale of the combustion wave, i.e. to study the evolution of the size of the liquid melt layer which may occupy only a part of the product region. We show, in particular, that a transition to relaxation oscillations may occur closer to the threshold of instability than in gasless solid fuel combustion. Our numerical and analytical results are in qualitative agreement."



A paper entitled "Propagation and Extinction of Forced Opposed Flow Smolder Waves," by A.C. Fernandez-Pello, B.J. Matkowsky, D.A. Schult, and V.A. Volpert is submitted to Combustion and Flame. The abstract of the paper reads: "Smoldering is a slow combustion process in a porous medium in which heat is released by oxidation of the solid. If the material is sufficiently porous to allow the oxidizer to easily filter through the pores, a smolder wave can propagate through the interior of the solid. We consider samples closed to the surrounding environment except at the ends, with gas forced into the sample through one of the ends. A smolder wave is initiated at the other end and propagates in a direction opposite to the flow of the oxidizer. We employ large activation energy asymptotic methods to find uniformly propagating, planar smolder wave solutions. We determine their propagation velocity, burning temperature, final degree of fuel conversion, and extinction limits. We also determine spatial profiles of gas flux, oxidizer concentration, temperature, and degree of conversion of the solid, including the burning temperature and final degree of conversion."

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-55

RESPONSIBLE CENTER: LeRC

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*Combustion of PTFE: The Effect of Gravity on the Production of Ultrafine Particles Generation*

PRINCIPAL INVESTIGATOR: Prof. J. T. McKinnon

Colorado School of Mines

CO-INVESTIGATORS:

P.W. Todd

University of Colorado

**TASK OBJECTIVE:**

This project is a study of the fundamental chemical and physical mechanisms of the production of ultra fine particles, and the nature of the particles themselves, generated by the thermal breakdown of polytetrafluoroethylene (PTFE) wire insulation. PTFE is a material that normally resists ignition and fire spread, but it can degrade when stressed by thermal and electrical overloads to release ultra fine (10 to 100 nm) particles as aerosols or smoke.

**TASK DESCRIPTION:**

The experimental study will be performed in three tasks, as follows:

1. Initial normal gravity tests, conducted at the Colorado School of Mines in a simplified apparatus, heat reference perfluoroalkane compounds in a tube furnace to generate ultra fine particles. These preliminary tests determine the effects of temperature and residence time on particle generation and develop the means for particle capture and gas analysis.
2. Subsequent normal gravity tests, also conducted at the Colorado School of Mines in a closed chamber apparatus, heat copper wires coated with PTFE. These advanced tests enable the detailed determination of particle sizes and concentrations by nonperturbing light scattering measurements and the collection of particles by thermophoretic sampling.
3. Subsequent microgravity tests, conducted by the investigators in the NASA Lewis Research Center drop tower and airplane facilities, are essential for an understanding of the effect of the low convection environment on the production of ultra fine particles and their possible dispersion and agglomeration, as a direct representation of potential hazard situations in spacecraft.

The accompanying analytical study seeks to develop a comprehensive predictive model to predict ultra fine particle production given inputs of heating rate, polymer quantity, degree of forced convection, and other factors. This analysis is based on elementary reaction rates from established databases, the investigator's studies on soot formation, and the interpretations of the results of the experimental tests.

**TASK SIGNIFICANCE:**

This study will examine the phenomenon of ultra-fine particle generation by the thermal degradation of a class of spacecraft wire insulation. The results contribute to the knowledge of the physical and chemical mechanisms of this process and offer practical applications in the reduction of health hazards in spacecraft and in the early warning detection of fire incidents by atmospheric smoke sampling.

**PROGRESS DURING FY 1994:**

This project began on June 27, 1994 with the award of NASA Grant NAG 3-1628. The initial task is to design the procedures of normal-gravity tests to determine the effects of temperature and resident time on the effluents produced by a pyrolyzing polymer and to develop a means of effluent gas analysis and particle capture. The original plan was to investigate the degradation of reference monomeric compounds, perfluoroalkanes, in a simple tube furnace. Instead, the studies initiated with investigation of the thermal reactions of the target polymer, polytetrafluoroethylene (PTFE), since there is an immediate application for detailed analyses of the products of this

material. Concurrent life-science studies at the University of Rochester Medical School, supported by a NASA Center for Research and Testing, shows that toxic solid and gaseous products are generated by pyrolytic reactions of PTFE at temperatures of 410 C and above. Gaseous products evolved in the tube-furnace tests will be separated and identified by gas chromatography and mass spectroscopy. Ultrafine-particle products will be collected on a simple filter plate, with morphological- and chemical-analysis methods to be determined.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 5/94 EXPIRATION: 4/98

PROJECT IDENTIFICATION: 962-22-05-64

NASA CONTRACT No.:NAG3-1628

RESPONSIBLE CENTER: LeRC

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*Premixed Turbulent Flame Propagation in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Suresh Menon

Georgia Institute of Technology

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The overall objective of this research is to characterize the behavior of turbulent premixed flames and to use the experimental data to validate a novel computational method to simulate accurately the behavior of premixed flames. In order to understand the behavior of turbulent flames, access to all the length scales (from the device size to the Kolmogorov scale) in the flow field is required. A possible method to achieve this is to reduce the flow velocity. However, in normal-gravity this is not possible due to gravitational acceleration and the turbulent stresses are overwhelmed by buoyant stresses. Therefore, experiments in microgravity will be carried out that will allow the study of low-speed turbulent flames without buoyancy effects.

**TASK DESCRIPTION:**

Premixed combustion of hydrogen-air mixtures will be studied in a Couette flow configuration which is essentially a flow between two parallel plates moving opposite to each other. The turbulent flow field is characterized with the flame speed being the primary variable of interest. Additional data on pressure rise and temperature is also measured.

**TASK SIGNIFICANCE:**

Improved understanding and modeling of practical turbulent flames will lead to increased efficiency and reduced pollutant formation. Practical applications of technology to industrial combustors and high throughput engines may be anticipated.

**PROGRESS DURING FY 1994:**

Preliminary test plans and schedules were prepared and presented to NASA LeRC drop tower and diagnostics personnel.

A detailed literature search was completed to determine what has been done related to premixed combustion and turbulent flows in Couette-type configuration. Relevant papers were collected and reviewed. The review of the literature clearly showed the following: (a) there has been no numerical and/or experimental work carried out for turbulent combustion (both premixed and non-premixed) in Couette flow configuration, (b) there have been quite a few experimental studies of plane-Couette flows, however, the configurations were quite different - in most cases, it consisted of only one wall moving and hence simulated a plane shear flow in a channel. These configurations were quite large setups and not considered feasible for the present study. Only two cases were found (other than the original report from Germany), which involved setups and testing procedure relevant for the present study. In particular, one configuration appears quite promising to study Couette cold flows since it appears to be easily constructed and will allow very long testing times. Cold flow testing will be the first experimental study since it would allow us to resolve the various issues related to turbulence measurements in such flows. (c) there have been numerous numerical studies of turbulent Couette flows; however, most of the studies were for the mean (steady-state) flow calculations using Reynolds-Averaged methods with turbulent closure and no unsteady flows have been studied. In summary, the review of available literature clearly indicates that there have been no studies of premixed combustion in turbulent Couette flows, and it further indicates even cold (non-reacting) unsteady flows have not been studied.

Based on the literature study, a numerical study of premixed combustion in a plane Couette Flow was initiated. The configuration is similar to the one planned for the experimental study. Premixed flame propagation is modeled using

the G-equation model. This approach allows the study of thin flames without requiring detailed kinetics modeling and therefore, is computationally very efficient. Unsteady turbulent flow is simulated using the large-eddy simulation methodology. The first phase involves simulations in two dimensions; however, we plan to move to full 3D within the immediate future. The 2D code is already operational.

A test problem was identified for the numerical code validation. This problem consisted of only one moving wall; however, detailed experimental data had been obtained for this case and therefore, the numerical predictions can be validated using the experimental data. Preliminary calculations of laminar Couette flows were carried out and an excellent agreement with the classical results was obtained. Currently, simulations of the turbulent flow are underway by using a turbulent inflow that consists of a mean flow and isotropic turbulence. The above noted studies are being carried out using a 2D LES code. However, the eventual goal is to simulate full 3D flows; therefore, a 3D version of this code is also being set up and is planned to repeat the 2D validation studies with the 3D code.

On the experimental side, the design issues for the cold flow experiments are being addressed. The exact dimensions have not yet been finalized, but the configuration under current consideration is similar to the setup used by Tillmark and Alfredsson in Sweden. The main differences are (a) the present study will employ gases (e.g., air and helium) and therefore, leakage is a serious issue, (b) the configuration will be much smaller overall in order to be applicable to the reacting Couette flow configuration, (c) the range of spacing between the two belts and the range of Reynolds number will be different and (d) it is planned to carry out detailed turbulence measurements and flow visualization of a no-heat release "flame" front (which will be generated by introducing in the channel center a new species that can be tracked - - currently acetone and byacetyl dye are being looked at).

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 5/94 EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-48

RESPONSIBLE CENTER: LeRC

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*A Fundamental Study of the Combustion Syntheses of Ceramic-Metal Composite Materials Under Microgravity Conditions*

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PRINCIPAL INVESTIGATOR: Prof. John J. Moore

Colorado School of Mines

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective is to obtain an improved understanding of the effect of gravity on the combustion synthesis of ceramic matrix-metal infiltrated composites and to develop new and improved-property materials.

**TASK DESCRIPTION:**

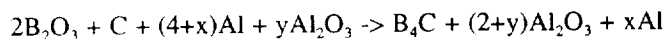
Combustion synthesis reactions are conducted under normal gravity (various orientations of samples with respect to gravity) and in microgravity to generate either porous or dense ceramic matrices in the presence of varying amounts of excess  $\text{Al}_2\text{O}_3$  (diluent) and/or excess aluminum, which will be available to fill residual pores by capillary action. The effects of gas generation during reactions on the porous matrix formation are studied under different levels of gravity. The dense composite materials are produced using a one step, low cost, simultaneous combustion-consolidated process. The microstructure and properties of both dense and porous product composite materials are also characterized.

**TASK SIGNIFICANCE:**

An improved understanding of the role of gravity on the combustion synthesis of ceramic-ceramic and ceramic matrix-metal composite materials allows for new approaches for the development of specified micro-structures, thereby resulting in improved material properties. One can also explore ways to develop brand new materials by microgravity processing (e.g. high surface area expanded or foamed ceramics, and fully dense interpenetrating phase composites). The expected benefits of such inexpensive, light, strong, and either highly porous or dense composite materials range from construction-support systems to liquid-metal, filtering-systems applications.

**PROGRESS DURING FY1994:**

The  $\text{B}_4\text{C}/\text{Al}_2\text{O}_3$  system using the reaction:



is being pursued based on the observation that this system showed expansions of approximately 150% under 2-g, and over 400% under 0-g conditions (Lear Jet), as opposed to 300% under n-g, after the combustion synthesis process. A new student will concentrate her efforts on the more accurate control of the combustion temperature to avoid melting  $\text{Al}_2\text{O}_3$ . If successful, this would provide an increased surface area in the expanded ceramic composite.

Two modes of combustion are being studied: 1) simultaneous, where the mixture is ignited by heating the whole pellet at once, and 2) propagating, where the ignition is at one end of the pellet. Depending on the value of y and combustion mode, the reactions are observed to be quenching, unstable, or stable, and the product has different pore sizes, distribution and expansion.

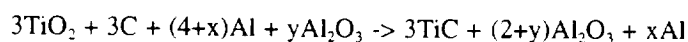
By using the 'simultaneous' combustion mode for cylindrical pellets they observed radial expansions of about 13% in addition to an axial expansion of about 15%, i.e. near three-dimensional uniform expansion. The scanning electron microscopy analysis shows that the samples contained a marked amount of submicron diameter whiskers.

Upon an informal review of the grant, the Space Experiments Division at LeRC came up with a number of recommendations. Prof. Moore agreed that a student should re-analyze the x-ray radiography tapes from previous NASA Lear Jet flights for a better verification of reaction speed and quantification of pore formation and progression in samples of the B<sub>4</sub>C/Al<sub>2</sub>O<sub>3</sub> system. X-ray and scanning electron microscopy (SEM) work is providing evidence for the speculation that reactions are occurring ahead of the combustion front.

Samples for the B<sub>4</sub>C/Al<sub>2</sub>O<sub>3</sub> system with 20 wt% excess Al are still planned to be flown on the Lear Jet with operation by LeRC personnel for about eight trajectories. After the 10 wt% excess Al<sub>2</sub>O<sub>3</sub> (diluent) samples, the student reacted some 5 wt% and no excess Al<sub>2</sub>O<sub>3</sub> samples in argon gas (inert atmosphere). Their SEM images also show the formation of shells within the sample. She will determine the interior structure and composition of the shells by using electron back-scatter imaging. She also plans to measure the surface area. A trend of continuous reduction in combustion temperature with increasing diluent is being observed.

The vacuum pump is in place to complete reactions utilizing 25 wt% Al<sub>2</sub>O<sub>3</sub> as a diluent. The goal is to decrease the combustion temperature of the reaction system below the melting point of Al<sub>2</sub>O<sub>3</sub> so that the reacted samples exhibit a higher surface area/weight ratio.

The other graduate student is processing the TiC/Al<sub>2</sub>O<sub>3</sub>/Al system using the reaction:



under normal gravity conditions using a hot press. He reports that the same level of uniformity of the metal network readily obtained under micro-g conditions would require pressures of 4000psi under normal-g conditions. He will also develop physical and computer models to predict these phenomena. He is studying the effect of green powder particle size on the kinetics, stability, and product microstructure of the TiC/Al<sub>2</sub>O<sub>3</sub>/Al system. Larger particles decrease the stability of the reaction. He has determined that metallic Al bonding between particles in products when excess Al is used is the main reason for the enhancement of strength and toughness. While fine particles result in more homogeneous pores in the final product, they produce no whiskers. On the other hand, coarse particles can produce whiskers when excess Al ( $x > 2$ ) is used.

The student then has achieved fracture toughness values twice as much as that of pure Al<sub>2</sub>O<sub>3</sub> using various values of the stoichiometry ( $x$  and  $y$ ), and consolidation pressure. The samples with higher fracture toughnesses exhibit smaller grain sizes and more homogeneous morphologies than their lower fracture toughness counterparts. Both Al<sub>2</sub>O<sub>3</sub> and Al are used in excess to control the combustion temperatures and, thereby, the microstructures and mechanical properties.

Finally, he has successfully defended his PhD Thesis in June 1994 and will continue working on the same system as a post-doc for six more months.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 8/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-22-05-38

RESPONSIBLE CENTER: LeRC

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*Flow and Ambient Atmosphere Effects on Flame Spread at Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Paul D. Ronney

University of Southern California

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## CO-INVESTIGATORS:

Sandra Olson

NASA Lewis Research Center (LeRC)

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**TASK OBJECTIVE:**

This program is a three-year experimental and theoretical study of the effects of ambient atmosphere on the properties of flame spread over thin and thick solid fuel beds. In particular the effect of the type of inert gas, which affects the Lewis numbers of fuel and oxidant, and the effect of the addition of sub-flammability-limit concentrations of gaseous fuels to the oxidizing atmosphere will be studied. The effect of convection will be studied through one-g and mg experiments with and without a forced flow. Moreover, the influence of thermal radiation, whose effect is known to be markedly different depending on the convection level, will be addressed.

**TASK DESCRIPTION:**

The emphasis of this study is on thermally thin fuels because of the limited  $\mu g$  test time available in ground-based facilities, and preliminary scaling analyses suggest that thermally thick fuels can be examined as well when gaseous fuel is added to the oxidizing atmosphere.

The experiments will be conducted in a combustion chamber in which a convective flow of a few cm/sec can be imposed in the direction opposite the flame spread. The oxidizing atmosphere will be mixed by the partial pressure method. For tests of Lewis number effects, inert gases He, Ne, N<sub>2</sub>, CO<sub>2</sub> and SF<sub>6</sub> will be used since they provide Lewis numbers from about 0.3 to 1.4. CO and CH<sub>4</sub> will be used for the gaseous fuels. Thin fuel samples will be ashless filter paper and thick fuel samples will be PMMA. Fuel samples of varying thickness will be ignited by the heat generated by a current passed through a coiled nichrome wire coated with nitrocellulose.

The primary diagnostics are video and an array of fine-wire thermocouples to measure the temperature simultaneously at several locations. The video records provide information on the spread rate and flame shape. The thermocouples give an independent check of the spread rates and the existence (or lack thereof) of a separate flame front in the case of added gaseous fuel. The temperature data may also be used to determine the heat flux from the gas phase to the fuel bed, which can be related to the spread rate.

**TASK SIGNIFICANCE:**

The understanding and control of accidental fires is a critical safety issue in both terrestrial and space-borne environments. The proposed work would provide insight that could be used to assess the fire hazards associated with non-standard atmospheres that might be employed in future manned spacecraft. Also, fires in enclosures produce a considerable amount of unburned vaporized fuel and partially combusted gases such as CO. One-g experiments have shown that the addition of combustible gases such as CO to the oxidizing atmosphere may increase the flame spread rate substantially. This study could provide information to improve models of fire development and spread in enclosures at one-g and  $\mu g$ .

The influence of weak forced convection is particularly important for studies of flame spread at  $\mu g$  because there is very little buoyancy-induced flow at  $\mu g$ . Experiments by Olson and collaborators show that the presence of forced convection currents (for example due to ventilation systems in manned spacecraft) can have a profound effect on the spread rate and extinction conditions. Consequently, the understanding of these effects is critical to understanding how fires might start, spread, and be extinguished at  $\mu g$  conditions.

**PROGRESS DURING FY 1994:**

To improve the sensitivity of the video imaging system for the very weak near-extinction flames, a shearing interferometer has been designed and is being constructed for evaluation. The shearing interferometer has no parts that have critical alignment requirements, and thus may be especially suitable for drop tests. The interferometric measurements may also be useful to supplement the thermocouple temperature measurements.

A graduate student is modifying our existing one-g flame spread apparatus for use in drop towers with regards to fuel sample mounting, optical access, and data acquisition.

As a precursor to the experimental study, an analytical study has been initiated in conjunction with Dr. Mike Delichatsios of Factory Mutual Research Corporation in Norwood, MA. The goal of the work is to extend Dr. Delichatsios's previous (1986) exact solution of flame spread over a pyrolyzing fuel bed to consider the effects Lewis number, finite-rate chemistry, and gaseous fuel addition on flame spread rate. A preliminary theory of Lewis number effects on flame spread over thin fuel beds, including finite-rate chemistry, has been obtained and compared with prior one-g data from Zhang et al (1992).

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 5/94 EXPIRATION: 4/98

PROJECT IDENTIFICATION: 962-22-05-61

NASA CONTRACT NO.: NAG3-1611

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

Delichatsios, M.A. and Ronney, P.D. "Horizontal and lateral flame spread on solids: closure and diffusional Lewis number effects." Fall Technical Meeting, Combustion Institute, Eastern States Section, Clearwater Beach, FL, December 5-7, 1994.

Combustion Research

PRINCIPAL INVESTIGATOR: Dr. Howard D. Ross

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The task objective is to advance the understanding of fundamental combustion phenomena and/or processes that are affected by the presence or absence of gravity.

**TASK DESCRIPTION:**

The research approach is to provide for limited precursor studies by external investigators and for the engineering and fabrication of hardware needed to conduct in-house research and assist in the research efforts conducted on-site at LeRC in support of Code UG-sponsored Principal Investigators (PI's) and National Research Council (NRC) graduate student researchers. Funds for facility overhead charges are provided through separate Research and Technology Operations Plans (RTOP) resources.

Subtasks are funded in part by this task, or are included for completeness at the request of NASA Headquarters.

**TASK SIGNIFICANCE:**

Significance varies with the various research efforts, and therefore, more focused, effort-specific, statements are given below.

**PROGRESS DURING FY 1994:**

Funds were disbursed to support service contractors to perform technical analysis and assistance for selected ground-based microgravity combustion science research grants and cooperative agreements. In addition, a number of research activities were conducted by onsite staff, including NRC post-doctorate fellows and Graduate Student Research Program fellows, associated with both ground-based and flight projects. The objective, approach, significance, and progress for each of these research activities are described below.

**Combustion Synthesis of Fullerenes - J. Brooker**

**Objective:**

The objective of this research is to produce, collect, and quantify fullerenes synthesized during combustion under normal gravity and microgravity and compare the results relative to: C60 and C70 composition, C60 and C70 percent of soot, and C70/C60 molar ratio. In phase one, acetylene will be burned in a gas-jet premixed flame; in phase two, benzene will be studied.

**Description:**

Flames will be used to generate fullerenes in a sub-atmospheric combustion chamber. Normal gravity tests will be performed to determine conditions favorable for fullerene production and to define a suitable method of collection and species separation/identification. Subsequent experiments will employ the 2.2 Second Drop Tower and possibly aircraft.

**Significance:**

The suggested potential applications for fullerenes include superconductors, lubricants, catalysts, high energy fuels, polymers, and biomaterials. Some of the emerging applications that are currently under investigation include the growth of diamond films on surfaces for protective coatings and electronic industries, their use as optical limiters to protect sensors from intense radiation sources, incorporation of fullerenes into photoconducting polymers with applications in light detection and electrostatic imaging, and the possible use of fullerene derivatives as drugs to combat AIDS by interacting with the active site of HIV.

**Progress:**

A literature search on fullerene properties and methods of production began along with a study of combustion texts to

become familiar with the theory. Microsoft Project will be used to identifying tasks, resources, and milestones and track project success. In addition, a research concept diagram was developed identifying hardware requirements and volume constraints.

Design of the burner, vacuum system, collection system, and diagnostic pallet began. Various parts were identified for procurement/fabrication and orders were submitted. With the LeRC Chemistry Laboratory, we are determining the analytical equipment and procedures necessary to identify fullerenes and to separate them from the other combustion products.

### **Investigation of Burning Droplets of Azeotropic Mixtures - Dr. R. Colantonio**

#### **Objective:**

Non-ideal mixtures that exhibit a relative extremum in their vapor pressure and "volatility reversal" at a particular composition (azeotropic point) are classified as azeotropic mixtures. This investigation will observe the liquid droplet and flame histories and location of the azeotropic point for a number of binary azeotropic mixtures.

#### **Description:**

Droplet burning of azeotropic mixtures will be performed at 1-g and at the NASA 2.2 second Drop Tower. A demonstrated droplet deployment and ignition system will be used. The droplet deployment system consists of two deployment needles that merge together via rotary motors. A fuel dispenser will form the droplet between the two needles tips. Once the droplet is formed the rotary motors withdraws the needles. Two hot-wire hoops will be used to ignite the fuel droplet once the needles have been withdrawn and the droplet is suspended during reduced gravity.

Both a color CCD camera and a high-speed 16 mm camera will be used to observe the burning characteristics of the azeotropic fuel mixtures.

#### **Significance:**

Many hazardous liquid wastes (e.g. aromatics, polyhalogenates, alcohol's and hydrocarbons) can form azeotropes. The unique feature about the behavior of azeotropes is the modulating of relative volatilities of the components by suppressing gasification of the lower or higher boiling point beyond a critical composition. This feature is very important in the disposal/incineration of azeotropic mixtures and in the chemical distillation industry. Limited data exists on the burning of azeotropic droplet mixtures. Both reduced gravity and normal gravity experiments on burning azeotropic droplet mixtures will lead to fundamental understanding of the volatility reversal and burning characteristics.

#### **Progress:**

Experimental hardware was procured and is currently being assembled with testing tentatively scheduled for December 1994. Tetrachloroethene/propanol and ethanol/water mixtures are likely candidates for the first series of testing.

### **A Transient Model of Flame Spread Over Solid Fuel in Microgravity - Dr. P. Ferkul (NRC)**

#### **Objective:**

The objective of this effort is to develop an unsteady model of flame spread over solids. Initially thin fuels will be examined, but eventually flames spreading over thicker, more realistic fuels will be considered. (When the fuel has an appreciable thickness, the flame does not typically reach a steady state.) The effect of ignition on the flame spread process is sought. Also, the flammability boundary, as a function of oxygen percentage and flow velocity, is to be investigated. The detailed dynamics of flame extinction can be examined with this model.

There are several other related objectives. The first is the development of a good algorithm to handle the interface between the solid and gas phases at the fuel surface when the fuel gets appreciably thick. The changing geometry due to fuel consumption complicates the specification of boundary conditions. The second is the potential treatment of turbulence since some of these long flames may tend to become turbulent in the downstream region. The third is the identification of those cases where a "quasi-steady" solid formulation will be adequate, as opposed to those where the fully unsteady solid phase equations are necessary.

#### **Description:**

An existing steady-state computer code will be modified. This original program is based on the SIMPLER algorithm which is a general fluid flow solver. The full Navier-Stokes equations are used, together with conservation of energy and fuel and oxygen species. A second-order finite-rate chemical reaction is assumed to model the combustion. Radiation from the fuel surface serves as a heat loss mechanism. The Cray Y-MP at NASA LeRC will be used to compute, store, and process the large amount of data which will be produced.

### Significance:

The purpose of this effort is to both guide and predict related experimental work. Together with the experimental work, the model will provide fundamental insight into flame spread at microgravity. The work has direct application to spacecraft fire safety.

### Progress:

The details of the flow around the fuel burnout point were examined. This region is important since it serves as the flame stabilization point and will thus have implications on extinction. Several references of cold flow in the leading edge region of a semi-infinite flat plate were examined. One reference suggested that using "parabolic coordinates" instead of the primitive cartesian coordinates may better capture details at the leading edge.

Using this suggestion, the parabolic coordinate system has been defined. The coordinate transformation of the Navier-Stokes equations from cartesian to parabolic was performed, using the vector form of the unsteady, compressible Navier-Stokes equations. The equations for parabolic-cylindrical and parabolic-axisymmetric coordinates were derived.

### Venting Extinguishment Experiment - J. Goldmeier (GSRP)

#### Objective:

The G.S.R.P. Venting Extinguishment Experiment is examining the extinction of a solid diffusion flame at low pressures in reduced gravity. The experiments being conducted will yield data on the flammability of the fuel at various pressures, oxygen concentrations, and flow velocities. This data will provide information regarding the effectiveness of the depressurization process as a fire extinguishment technique in reduced gravity.

#### Description:

The Venting Extinguishment Experiment is examining the combustion and extinction behavior of a solid, horizontal, PMMA cylinder in a low-speed flow in reduced gravity. The depressurization process is being examined in three phases. The first portion involved 1-g testing to determine the appropriate test parameters. The second portion of the research examined the effect of reduced gravity on the selected combustion configuration utilizing the NASA Lewis 2.2 Second Drop Tower.<sup>1</sup> The third phase of the research will examine the effects of flow and depressurization on the flame in 1-g and in low-gravity. The low-g depressurization experiments will be conducted on board a NASA reduced gravity aircraft. (This research is part of J. Goldmeier's doctoral program at Case Western Reserve University).

#### Significance:

Combustion of solids in low-speed forced flows at reduced gravity is relevant to fire safety in spacecraft. In an emergency a compartment within a space vessel could be intentionally vented to space to extinguish a fire. However, the effect of the induced flow on the fire in the low gravity and low pressure environment is unknown.

#### Progress:

A series of tests were conducted on board the KC-135. In the combustion tests the PMMA cylinders (2.5 cm in length, 1.9 cm in diameter) were ignited during the 2-g portion of the trajectory with flow in the chamber. A focus of the analysis has been the centerline temperature of the samples.

Steady-state extinction tests (1g) were also conducted. In these tests a horizontal PMMA cylinder was ignited in a forced flow and the pressure was slowly reduced until flame extinction occurred. During the experiment the velocity was kept constant.

In these tests three different combustion modes were evident as the pressure was reduced. The first was a flame which surrounds the cylinder completely. The second mode of combustion occurred when the chamber pressure was reduced to approximately 0.1 atm. The flame at the lower stagnation point of the cylinder experienced blow-off, and the flame which was still burning along the upper surface of the cylinder began to oscillate. (This behavior was previously seen in 1g oxygen depletion experiments.) The third mode occurred after the oscillations stopped. The flame slowly transitioned into a wake flame. The flames along the upper surface extinguished at approximately 0.07 atm; this pressure seemed to be independent of the flow velocity.

During all tests, the temperature at the center of the sample was measured by a thermocouple probe. There was a correlation between the measured solid temperature at the center of the cylinder pressure at extinction. As the solid temperature increased, the pressure required to extinguish the flame decreased.

A series of reduced gravity tests were conducted again on board the NASA Lewis Lear Jet. Forty-four trajectories were accomplished in nine flights. The experiments examined the steady-state extinction with a constant forced flow in reduced gravity. The chamber pressure and the temperature at the center of the solid were measured during these experiments.

Above a critical solid centerline temperature the flames did not extinguish in reduced gravity. Examining the trend in that data indicated that the pressure required to obtain extinction decreased as this critical temperature increased. At temperatures below this range, the flames extinguished during reduced gravity. This has implications for the possible use of venting (depressurization) as a method for extinguishing fires on board spacecraft.

**Heat Release Effects on Shear-Layer Instabilities - Dr. U. Hegde****Progress:**

During FY 94, studies were conducted in primarily two areas:

**(i) Heat release effects on shear-layer instabilities**

The effects of unsteady heat addition on instabilities of parallel shear layers in the absence of gravity was analyzed. It was shown that, in the linear regime, the influence of heat addition is felt only through the time-averaged velocity and temperature fields, that is, the unsteady component of the heat addition plays no active role. The analysis which has application to both premixed and diffusion flames was published in AIAA Journal.

**(ii) Effects of unsteady heat transfer to a burning solid fuel**

In collaboration with Dr. M. Vedha-Nayagam, analysis of the oscillatory behavior of opposed-jet diffusion flames was extended to a stagnation point solid fuel diffusion flame. This is an area where microgravity environment may be of use in studying the effects of turbulence on flames. The effects of velocity fluctuations in the oxidizer stream on oscillations in the burning rate of the fuel and flame intensity were investigated. The analysis was carried out to second order to investigate the time-averaged shift away from the undisturbed flame configuration. It was found, for cases considered, that the velocity oscillations caused an increase in the flux of reactants into the flame. Results were presented at the Central States Section Meeting in June.

**Diffusion Flame Extinction Dynamics - V. Nayagam****Objective:**

The objective of this research is to investigate the extinction dynamics of diffusion flames at small, as well as large stretch rates. The experimental portion of this study employs a rotating fuel-disk. The diffusion flame supported by the spinning fuel disk is embedded in the induced 'Von Karman' boundary layer. This novel flow configuration enables one to control the stretch rates precisely. The theoretical effort makes use of large activation energy asymptotics to examine the extinction process. The attempt is to provide a unified of high stretch extinction (blow-off) and radiation heat loss dominated low stretch extinction.

**Significance:**

A knowledge of conditions under which diffusion flames extinguish is of fundamental as well as practical importance. Furthermore, the concept of laminar flamelets as an embedded structure that controls turbulent diffusion flames has provided a new impetus to further understand laminar extinction conditions in a variety of flow configurations. In the present study a rotating fuel disk induces a three dimensional boundary layer in which a laminar diffusion flame is established. A range of chemical and fluid mechanical time scales over which extinction conditions occur are produced by changing the environmental conditions (pressure and oxygen concentrations), fuel, and the spinning velocity of the fuel disk. High speed filming of the burning process, and temperature measurements provide quantitative information regarding the extinction dynamics.

**Progress:**

The large experimental apparatus used earlier for normal gravity studies has been redesigned and miniaturized to fit into the standard drop-tower frames. The new design is currently being built. A theoretical model for the extinction process using the activation energy asymptotic technique has been developed. The model predicts extinction at high stretch rates (high RPM) as well as radiation heat loss induced low stretch extinction. The extinction condition is characterized by a critical Damkohler number.

**Stability of Laminar Premixed Gas Flames - Dr. H. Pearlman (NRC)****Objective:**

The objective of this study is to develop a fundamental understanding of the inherent stability of premixed gas flames at earth and micro-gravity.

### Description:

The research approach is to design and construct hardware used to study freely-propagating flames in tubes in both normal and microgravity. This experimental configuration is chosen because it eliminates the additional complexities of heat loss and hydrodynamic effects common to burner stabilized flames.

### Significance:

Understanding the intrinsic stability of premixed gas flames is essential to our fundamental understanding of heat and mass transport in the presence of chemical reaction. This work is applicable to our understanding of combustion phenomena as well as to our knowledge of a broader class of chemical and biological systems known as reactive-diffusive systems, which include such diverse phenomena as the patterns of spots on animals, the spread of infectious disease, the formation of spiral galaxies, as well as the rhythmic beating of a human heart and the solitary voltage pulses which travel along nerve fibers.

### Progress:

One of the most fundamental problems in premixed gas combustion is the stability of a steadily-propagating, planar flame in a quiescent environment in the absence of forced or natural convection (buoyancy). Its stability will depend on: (1) the rate at which the heat liberated by the flame diffuses into the cold reactants versus the rate at which the stoichiometrically scarce reactant, the rate-limiting component, diffuses into the flame (i.e., the diffusive-thermal mechanism) and (2) the ratio of the heat lost to the heat generated by the flame. The first of these two effects can be characterized by the Lewis number ( $Le$ ) of the mixture where the  $Le$  is defined as the ratio of the thermal diffusivity of the bulk mixture to the mass diffusivity of the stoichiometrically scarce reactant in the bulk mixture.

Low- $Le$  (cellular flame) instabilities have undergone extensive experimental and theoretical treatment during the past century, yet, the high- $Le$  instability has managed to evade experimentalists, until now, even though theoretical treatment by Joulin and Clavin, Matkowsky and Olagunju, and Booty et. al. have strongly suggested that pulsating and/or traveling wave instabilities arise in high  $Le$  number mixtures.

The first observations of these instabilities were made in the 2.2 second drop tower at NASA LeRC using a lean mixture of butane and oxygen diluted with helium ( $Le_{3.0}$ )<sup>1,2</sup>. Based on these tests, we realized that the reason the instabilities have not been observed earlier is because the oscillation frequency is on the order of 100 Hz (or higher); much too fast to be detected by the human eye or at standard video framing rates.

Consequently, we use a high speed intensified video camera to visualize the flames. We observe two intrinsically unstable modes of flame propagation: (1) a pure radial pulsation and (2) a combined radial pulsation and traveling wave instability. These modes have been observed at  $lg$  and  $ug$  which suggests that the instabilities are not buoyantly-induced, but rather inherent to the flame itself, caused by an imbalance between heat and mass transport in the presence of chemical reaction.

One of the most striking features of these high  $Le$  number instabilities is that the spatio-temporal patterns rival those observed in other excitable media. Specifically, this premixed gas-phase reaction exhibits many of the same features and analogous dynamical behavior as numerous biological and chemical systems including the well-studied, liquid-phase Belousov-Zhabotinskii (BZ) reaction. Both these premixed gas flames and the BZ reaction exhibit chemical fronts which spontaneously develop from single or multiple pacemaker sites into circular ring wave(s) or rotating spiral wave(s). This is indicative of self-propagating waves of chemical activity in excitable media believed to affect such diverse phenomena as the patterns of spots on animals, the spread of infectious disease, the formation of spiral galaxies, as well as the rhythmic beating of a human heart and the solitary voltage pulses which travel along nerve fibers.

### Radiative Flame Extinction at Large Droplet Radius - P. Struk (GSRP)

#### Objective:

To verify experimentally the existence of a limiting large droplet radius above which a spherical steady flame cannot be sustained due to gas-phase radiative loss. Large fuel droplets are simulated using a wetted porous sphere continuously supplied fuel by a syringe pump.

#### Description:

The research consists of two phases: a ground-based phase and a microgravity phase. The ground-based phase involves the development of porous spheres, hardware buildup, as well as ascertaining the feasibility and techniques of using porous spheres to simulate droplet burning. In the microgravity phase, wetted porous spheres are burnt in various atmospheric conditions to ascertain a droplet flammability map based on droplet diameter. In microgravity, the radius of the droplet is varied by adjusting the fuel flow rate to the porous sphere via the syringe pump.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

### Significance:

The verification of a limiting large droplet radius will contribute to our understanding of droplet burning and extinction, a fundamental topic in combustion. It will also support the premise that gas-phase radiative loss alone can cause diffusion flame extinction in  $\mu\text{g}$ .

### FY94 Progress:

Hardware was designed, selected, and assembled. Two sets of experiments were performed during the ground-based phase in normal gravity to demonstrate the feasibility of simulating fuel droplets using a wetted porous sphere. In both sets of experiments, the fuel used was n-decane. The first set of experiments were performed at atmospheric pressure and used a porous bronze sphere, 5 mm in diameter. A wetted fuel surface was not observed during an entire burn due to an excessive heat feedback from the flame predominately due to the convective environment. The heat feedback, in turn, causes a high fuel consumption rate which the syringe pump could not supply.

A second set of experiments were performed at roughly 1/3 atmospheric pressure and used a 4 mm porous bronze sphere. A wetted fuel surface was observed during an entire burn at these conditions. Reducing the pressure decreased the mass burning rate of the fuel (via chemical kinetics). At reduced pressure, the syringe pump could provide fuel to the porous sphere at a rate equal to or greater than the consumption rate of the fuel. These results indicate the feasibility of controlling the droplet radius in microgravity where convective effects are significantly reduced.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 1/91    EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-22-05-08

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Ferkul, P.V. and T'ien, J.S. A model of low-speed concurrent flow flame spread over a thin fuel. Combustion Science and Technology (in print), (1994).

Hegde, U. Heat release effects on the instability of parallel shear layers. AIAA Journal (accepted), vol. 31 no. 1, 206 (January 1994).

#### Presentations

Vedha-Nayagam, M. and Hegde, U. "Effects of free stream oscillations on heat transfer to a burning fuel surface." Spring Meeting of the Combustion Institute, Central States Section, June 1994.



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*Combustion of Solid Fuel in Very Low Speed Oxygen Streams*

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PRINCIPAL INVESTIGATOR: Dr. Kurt R. Sacksteder

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this program is to obtain experimental experience and data to improve the understanding of the mechanisms that control the spreading of flames and the flammability of materials in low-speed oxidizing flows, including buoyantly-driven and forced concurrent flows and buoyantly-driven opposed flows.

**TASK DESCRIPTION:**

New and unique experimental apparatus are to be developed to obtain flame spreading observations in partial gravity and in forced flows in microgravity. Partial gravity tests are to be conducted aboard NASA research aircraft flying parabolic trajectories, altered from the traditional condition of near free fall, to obtain the desired reduced levels of acceleration. Low-speed forced flows are to be obtained in microgravity drop towers using mechanical devices to establish purely-forced (non-buoyant) flows. In each instance, detailed measurements of flame ignition, spreading, and limiting behavior are to be made.

**TASK SIGNIFICANCE:**

This work attempts to provide a fundamental understanding of the practical flame-spreading environment of low-speed flows including: purely buoyant flows in partial gravity (between microgravity and normal gravity), and purely forced flows only possible in microgravity. In normal gravity, buoyancy forces induce flows in spreading flames having velocities of no less than 20-30 cm/sec. Interactions between flames and lower speed flows, therefore, cannot be observed in normal gravity. In the low-speed flow regime, flow velocities approaching the spreading velocity of the flames can be examined. These flames are expected to demonstrate entirely new spread-rate limiting mechanisms, different than the normal-gravity counterpart. These tests are also expected to demonstrate lower flammability limits of solid fuels than any observed in normal gravity. Additionally, this work will attempt to distinguish between influences of buoyantly induced flows and flows of similar intensity that are externally imposed.

**PROGRESS DURING FY 1994:**

This task was completed during fiscal year 1994 with the award of a new flight experiment project entitled, "Combustion of Solid Fuel in Very Low Speed Oxygen Streams," under NRA-93-OLMSA-1, "Microgravity Combustion Science: Research and Flight Experiment Opportunities."

At the transition to the flight program, this task has achieved the following major accomplishments:

1. A comprehensive testing program of flame spreading and flammability limit determinations for a thin fuel burning in low-speed forced-concurrent flows has been conducted. These experiments include drop tower experiments in a microgravity wind tunnel, and experiments in a unique experimental device, developed under this program, with which the lowest speed flows are obtained by translating the burning sample through the oxidizing environment. In these experiments a low-speed flammability limit has been observed for forced flows at about 12% oxygen atmospheric mole fraction (normal air is 21% oxygen), far below the opposed-flow limit observed in microgravity of about 15% oxygen. Flame spread rates were measured over a wide range of oxygen environments and flow velocities. All forced-flow results were dominated by the ignition process because of the limited testing time available in the ground-based facilities.

2. A comprehensive testing program of flame spreading and flammability limit determinations for a thin fuel burning upward in purely buoyant (concurrent) flows has been conducted. These experiments were conducted in an aircraft facility operating under partial-gravity conditions. These experiments have demonstrated reduced-gravity upward-spreading flammability limits similar to those observed in the low-speed forced flow experiments, and similar to the normal gravity upward spreading limit, all approximately 12% oxygen. At the reduced-gravity levels obtainable in aircraft testing, (between 0.05 and 0.6 times normal Earth Gravity) the buoyant velocities are sufficiently high that flammability limit and flame spread behavior are similar to normal gravity behavior, ie. finite rate chemical reactions limited by reactant residence times. At these relatively high speed flows (even at 0.05 g) flame spread rates are unsteady, because of either inherent unsteadiness or ignition effects. Until lower levels of partial-gravity are made available, a transition to "low-but-not-zero" gravity behavior in concurrent flow flame spreading cannot be observed.

3. A comprehensive testing program of flame spreading and flammability limit determinations for a thin fuel burning downward in purely buoyant (opposed) flows have been conducted. These experiments were conducted in an aircraft facility operating under partial-gravity conditions. These experiments have demonstrated increases in flammability as the gravitational acceleration is reduced from normal Earth gravity, to a low of between 13% and 14% oxygen near 0.05 g compared to the normal gravity limit of 15.7% oxygen. Thus, in the case of downward flame spreading, Lunar and Martian gravity levels present a greater fire hazard than observed on Earth. Flame spread rates also increase with reductions in acceleration from normal gravity, peak at some intermediate gravity level then decrease with further reductions in gravity. In normal air (21% oxygen), these peak spread rates occur in the vicinity of Lunar and Martian gravity levels.

4. Combining the results of items 2) and 3), this task has provided the first comprehensive measurements of flame spread behavior of a single material over the full range of flow conditions from "high-speed" flows in the concurrent direction, through low-speed flows in reduced gravity (transitioning from concurrent to opposed flow), to "high-speed" flows in the opposed direction.

5. Based upon the results of the forced concurrent flow testing, a successful proposal was submitted for the Spacelab/Middeck/Mir Glovebox Experiment program entitled, "Forced Flow Flamespread Test."

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	2
MS Students:	1	MS Degrees:	2
PhD Students:	1	PhD Degrees:	0

**TASK INITIATION:** 5/89 **EXPIRATION:** 2/94

**PROJECT IDENTIFICATION:** 962-22-05-28

**RESPONSIBLE CENTER:** LeRC

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**Journals**

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**Presentations**

Sacksteder, K.R. and T'ien, J.S. "Downward diffusion flame spread and extinction in variable gravitational fields: Lunar and martian simulations." presented to the AIAA 31st Aerospace Sciences Meeting and Exhibit, AIAA-93-0828. 1993.

*Reduced Gravity Combustion with 2-Component Miscible Droplets*

PRINCIPAL INVESTIGATOR: Prof. Benjamin D. Shaw

University of California, Davis

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective is to improve understanding of the combustion of two-component miscible droplets in reduced gravity. We will consider both experimental and theoretical aspects of the combustion behaviors.

**TASK DESCRIPTION:**

Reduced-gravity experiments will involve burning two-component miscible droplets in environments of differing pressures and compositions. Data will be obtained by direct photography of droplets and flames. Droplets with components that differ significantly in volatility and which are initially in the millimeter-size range will be studied. Data will be collected on the following: transient liquid behaviors (e.g., diameter histories and disruption), transient flame behaviors (e.g., sudden flame contraction and extinction), and sooting (e.g., luminosity and transport of observable soot particles).

Hydrocarbon (e.g., heptane/hexadecane) or alcohol mixtures in oxygen mole fractions from about 0.1 - 0.5 and initial pressures from 0.5 - 2 atm are to be used. Different inerts will be used in the gas phase to enhance or inhibit certain phenomena; e.g., CO<sub>2</sub> may inhibit soot formation, while helium may promote extinction by increasing gas transport rates. Theoretical and computational research in support of this program will also be undertaken. This research includes: modeling of fundamental droplet combustion phenomena; studies of allowable acceleration levels and durations to define experiment requirements; and studies of issues such as allowable droplet oscillation amplitudes, oscillation decay times, and pre-ignition vaporization; development of a new droplet combustion apparatus for use in the NASA Lewis 2.2-Second Drop Tower Facility was undertaken at the request of the cognizant NASA monitor after the original proposal was awarded.

**TASK SIGNIFICANCE:**

The reduced gravity experiments should yield insight into the efficiency of liquid-phase species diffusion, since this diffusion may markedly influence combustion behaviors (e.g., three-staged combustion behaviors). By noting times for flame contraction to occur, calculations for effective liquid-phase species diffusivities may be made (e.g., with asymptotic or with numerical models). Finally, unique sooting behaviors may appear since gas-phase compositions may differ significantly with time. Differences in sooting may be observable photographically by noting behaviors of apparent flame luminosities and soot particles large enough to be observed.

The theoretical efforts will aid interpretation of the reduced-gravity experiments as well as improve understanding of the fundamental phenomena that occur during droplet combustion in reduced-gravity.

**PROGRESS DURING FY 1994:**

Experimental:

A digital image analysis system has been constructed and used for analysis of films that were taken of preliminary experiments performed using a NASA-supplied drop apparatus. During fiscal year 93/94 a new drop apparatus was designed and built for use in the NASA Lewis 2.2 sec drop tower. The apparatus was constructed at UC Davis. The electronics on the apparatus, except for some modifications, are based on the design of a NASA apparatus. The mechanical design is completely new though it is based on knowledge and experience accumulated at NASA. The drop apparatus is now being used for experiments.

Experiments are being done with different initial environmental compositions and pressures. Initial oxygen mole fractions from about 0.1 to 0.5 and initial pressures from 0.5 to 2 atm are being used. Different inerts are used in the gas phase to enhance or inhibit certain phenomena. For example, helium may promote extinction and higher burning rates, while CO<sub>2</sub> may inhibit soot formation. Flame contractions and droplet disruptions have previously been observed for initially-pure heptane or decane droplets burning in low-gravity environments. These behaviors have been postulated to result from sooting effects. If this is correct, flame contractions and droplet disruptions arising from surface buildup of the less volatile component in a binary fuel droplet may be influenced to an unknown extent by sooting, making interpretation of experimental results difficult. Experiments are being performed to search for conditions where sooting appears to influence combustion negligibly.

#### Computational:

Detailed computational modeling (in collaboration with Dr. H. A. Dwyer) has focused upon predicting the effects of capillary flows (from droplet surface temperature and/or composition gradients) on droplet vaporization. Calculations of single-component droplet vaporization in a hot environment have clearly shown that surface-tension gradients can dramatically influence droplet vaporization and internal circulation behaviors under conditions that are representative of slowly-drifting droplets in reduced-gravity experiments as well as rapidly-translating droplets in practical high-pressure sprays. These calculations are now being extended to combustion of two-component miscible droplets, for which surface-tension gradient effects are expected to be especially significant.

#### Analytical:

To aid interpretation of experimental efforts, analytical studies of the behaviors of gasifying droplets in reduced-gravity environments are being performed. These efforts are focused on studying: (1) effects of small gravitational levels on droplet vaporization/combustion; and (2) hydrodynamic stability of spherically-symmetric vaporization/combustion of two-component droplets. Each effort is presently focused upon modeling evaporating droplets; combustion will be considered in the future. The gravity-level research is expected to provide scaling relations for the effects of small gravitational accelerations on vaporization rates and gas-phase structures. The stability analyses address the problem of hydrodynamic stability of a multicomponent mixture droplet during evaporation/combustion; specifically, the question is addressed as to whether spherically-symmetrical evaporation/combustion is hydrodynamically stable.

The studies of gravitational effects involve solving the equations for conservation of energy, mass and momentum (Navier-Stokes) using singular perturbation techniques. We are presently focusing on steady gravitational levels and stationary droplets, though oscillatory gravity levels and/or slowly drifting droplets will be studied in the future. The governing equations are made dimensionless and relevant dimensionless parameters are identified. Analysis shows that when gravity levels are small, an appropriate small parameter of expansion is a Richardson number ( $Ri = gR/U_s^2 = \text{buoyancy/momentum}$ ) based upon the droplet radius  $R$ , the radial gas velocity at the droplet surface  $U_s$ , and the gravitational acceleration  $g$ . Using this parameter it is shown that distinct spatial regions exist where different physical phenomena are dominant. Near a droplet, radial velocity gradients are sufficiently large such that gravitational effects are small, and in the first approximation the gas-phase solution can be considered to be spherically symmetric. Far away from the droplet, however, viscous and pressure-gradient terms in the momentum equation become of the same order as the buoyancy term. Different zones therefore exist, allowing techniques of matched asymptotic expansions to be used. This methodology is currently being followed for analysis of droplets vaporizing in hot atmospheres. When this analysis is completed, the calculations will be extended to droplet combustion.

As noted above, the analysis has demonstrated that for the case of droplet gasification in a hot environment, distinct spatial zones exist. Spherical symmetry prevails in the first approximation near a droplet. Far away from a droplet, however, gravity effects become important and a stagnation point is predicted to form in the flow for any nonzero gravity level regardless of its magnitude; the location of this stagnation point is predicted to scale as  $Ri^{(-1/3)}$ . The inner (spherically symmetric) zone and an outer zone that includes the stagnation point have been analyzed. Another zone exists further out where the flow appears to resemble a viscous plume driven by natural convection, for example similar to what would exist around a heated or cooled sphere. We are presently in the process of investigating this zone and matching the solution to the inner zones to provide a complete description of the flowfield and its influences on droplet gasification.

The hydrodynamic stability analysis was carried out using linear stability theory. The normal-mode method was used in the analysis. The disturbance equations for conservation of mass, momentum, energy and species in the liquid and gas phases were derived from the general governing equations, written in spherical coordinates, by introducing small disturbances. Furthermore, the disturbance equations were linearized and nondimensionalized. By assuming a certain type of solution and using the spherical harmonic decomposition together with the quasisteady assumption in the gas phase, the partial differential equations were transformed into a set of linear second order nonhomogeneous ordinary differential equations. By taking advantage of the special properties of the spherical harmonic decomposition for solenoidal velocity fields, the 3-dimensional problem was reduced to a 1-dimensional problem in the radial coordinate. The disturbance equations were solved using different methods. In the gas phase negligible terms were dropped from both the energy and species equations, simplifying them to standard nonhomogeneous Euler equations. In the liquid phase the boundary-layer nature of the problem in the species equation was identified, and consequently a solution using matched asymptotic expansions was adopted for this equation. The energy equation in the liquid phase was solved by using a truncated Frobenius series. For both phases the momentum equation was simplified to a biharmonic equation which could be solved analytically.

The interface conditions between the two phases were defined accounting for balances of mass, momentum and species, complemented with the equations accounting for tangential stresses, tangential velocities and temperature. Here again, use was made of the spherical harmonic decomposition representation and its special properties for solenoidal fields.

These efforts have allowed an analytical solution to be developed for the growth factor in the stability analysis. This solution is presently being investigated to find the marginal stability curve under various conditions. It has tentatively been found that certain binary mixtures are anticipated to be stable, while others may be unstable. Stability is influenced both by surface-tension gradients as well as by viscous stresses that occur from uneven surface vaporization rates.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	1
PhD Students:	2	PhD Degrees:	0

TASK INITIATION: 12/91    EXPIRATION: 3/95

PROJECT IDENTIFICATION: 962-22-05-39

NASA CONTRACT NO.: NCC3-245

RESPONSIBLE CENTER: LeRC

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BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Journals**

Niazmand, H., Shaw, B.D., Dwyer, H.A., and Aharon, I. Effects of Marangoni convection on transient droplet evaporation. Combustion Science and Technology, (accepted).

**Presentations**

Niazmand, H., Shaw, B.D. and Dwyer, H.A. "Effects of Marangoni convection on transient droplet evaporation in reduced gravity." Paper presented at the Fall Meeting of the American Chemical Society, Chicago, Ill. August 22-27, 1993.

Niazmand, H., Shaw, B.D. and Dwyer, H.A. "Effects of Marangoni convection on transient droplet evaporation at elevated pressures and with negligible buoyancy." Paper 93-083 presented at the 1993 fall meeting of the Western States Section of the Combustion Institute, SRI International, Menlo Park, CA, 1993.

*Quantitative Measurement of Molecular Oxygen in Microgravity Combustion*

PRINCIPAL INVESTIGATOR: Dr. Joel A. Silver

Southwest Sciences, Inc.

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

There are two objectives regarding this task. The first is to develop a diagnostics based on diode laser absorption spectroscopy for use in microgravity combustion research. The general technique is expected to be applicable to major gases involved in combustion and in spacecraft cabin environments. The specific application of this task is directed to the quantitative determination of molecular oxygen. The second objective is to develop an improved understanding of the relative roles of diffusion and reaction of oxygen in microgravity combustion. Oxygen has a major role in controlling flame properties such as flame front speed, extinguishment, flame size, and temperature.

**TASK DESCRIPTION:**

This investigation involves the determination of molecular oxygen using diode laser absorption spectroscopy. This technique uses a compact, low power optical system to probe the near infrared bands of oxygen. Though these bands are relatively weak, high sensitivity is maintained by means of wavelength modulation of the diode laser with detection at twice the modulation frequency. A compact laser scanner permits the laser beam to traverse the entire flame region. This scanner eliminates the need to use multiple optical fiber lines-of-sight in the setup. A compact electronics package includes all control, detection, and data acquisition electronics.

**TASK SIGNIFICANCE:**

Oxygen, as the primary oxidizer in flames, controls the major flame properties such as flame-spread, flame-front speed, temperature, and its extinguishing properties. Therefore, its quantitative determination is necessary for both an improved understanding of flame behavior in general, and for fire safety on board spacecraft. Oxygen measurement is also critical for ground-based applications such as engine efficiency, energy production, air quality monitoring, and industrial processes such as steel manufacturing. The specific technique under development is a compact, lightweight, and rugged instrument offering a wide range of laboratory, field, and industrial process measurements.

**PROGRESS DURING FY 1994:**

Optimum spectral lines for simultaneous measurement of temperature and O<sub>2</sub> concentration have been computed. Four pairs of lines have been identified which show appropriate temperature sensitivity. Expected temperature accuracy is  $\pm 25$  K. Optimum lasers for these lines have been ordered.

The PI is investigating the possibility of using Vertical Cavity Surface-Emitting Lasers (VCSEL's) as a potential replacement for the conventional laser diodes. The VCSEL's show promise of increased long-term reliability, wider tuning range, better beam profile, and reduced mode-hopping.

Optical and mechanical design has been completed and components have been ordered, received, or fabricated. Assembly is underway. Various lens types have been tested for laser focusing.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/94 EXPIRATION: 6/97

PROJECT IDENTIFICATION: 962-22-05-51

NASA CONTRACT No. NA53-26553

RESPONSIBLE CENTER: LeRC

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*Numerical Modeling of Flame-Balls in Fuel-Air Mixtures*

PRINCIPAL INVESTIGATOR: Prof. Mitchell D. Smooke

Yale University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of the proposed work is to understand if and the mechanisms by which stationary, spherical flames, sometimes called flame balls, stabilize in a quiescent, premixed gaseous fuel-oxidizer environment. These flames have been observed experimentally shortly after ignition in microgravity, but their long-term persistence is as yet undetermined.

**TASK DESCRIPTION:**

The approach to meet this objective is to modify an existing numerical model, which includes full chemical kinetics, to account for radiative heat losses. The predictions will then be compared to the experimental observations of Prof. P. Ronney as well as theoretical results developed by Prof. J. Buckmaster.

**TASK SIGNIFICANCE:**

The significance of this effort is to enhance the understanding of very basic combustion phenomena, such as: flammability limits and stabilization mechanisms, and the probability that it will find its way into combustion science textbooks.

**PROGRESS DURING FY 1994:**

The ignition of near limit hydrogen/air mixtures can lead to the formation of "flamecaps"--discrete curved flames small on the scale of the confinement vessel which rise because of buoyancy. Recently it has been shown that, in low gravity, when buoyancy effects are small, flameballs can be generated. Flameballs are stationary spherical structures whose existence appears to require the following ingredients: a near-limit mixture; small Lewis number; and radiative, convective, and conductive losses. Flameballs have been observed in a variety of mixtures. For example,  $H_2$ /air,  $H_2/O_2/CO_2$ ,  $H_2/O_2/SF_6$ , and  $CH_4/O_2/SF_6$ .

Numerical solutions of flameballs have been constructed for  $H_2$ /air mixtures using an accurate description of chemical kinetics, diffusive transport, and radiation losses. A previously-developed code has been modified for plane flames to account for the spherical geometry, radiation, and the absence of a flow field. A lean limit equivalence ratio of 0.0866 was predicted and a rich limit of 2.828. For any equivalence ratio between the two limits there are two solutions. One is characterized by a small flame, incomplete reactant consumption, and negligible radiation losses. The other is characterized by a large flame, complete consumption of one of the reactants, and significant radiation losses. The maximum temperature varies between 1200 and 900 K as the two solution branches are traversed. There is evidence that the kinetic mechanism adopted here is not as accurate as it might be at low flame temperatures. Also, there are some uncertainties in the present description of the radiation transport.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94 EXPIRATION: 4/98

PROJECT IDENTIFICATION: 962-22-05-60

RESPONSIBLE CENTER: LeRC



*Combustion of Solid Fuel in Very Low Speed Oxygen Streams*

PRINCIPAL INVESTIGATOR: Prof. James S. T'ien

Case Western Reserve University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this program is to develop a theoretical model to improve the understanding of the mechanisms that control the spreading of flames over solid fuels and the flammability of materials in low-speed oxidizing flows, including buoyantly driven concurrent and opposed flows and forced-concurrent flows.

**TASK DESCRIPTION:**

A numerical simulation of the concurrent-flow flame spreading process is to be developed that accounts for both the stabilization of the flame at the leading edge facing the flow and the downstream tip of the flame where the flame spreading occurs. The required computational domain must therefore be large in comparison to the opposed-flow case. A steady solution will be formulated and solved first, followed by a transient version. The model is to accommodate first thin and then thick fuels.

**TASK SIGNIFICANCE:**

The work attempts to provide a fundamental understanding of the practical flame-spreading environment of low speed flows including: purely buoyant flows in partial gravity (between microgravity and normal gravity), and purely forced flows only possible in microgravity. In normal gravity buoyancy forces induce flows in spreading flames having velocities of no less than 20-30 cm/sec. Interactions between flames and lower speed flows, therefore, cannot be observed in normal gravity. In the low speed flow regime, flow velocities approaching the spreading velocity of the flames can be examined. These flames are expected to demonstrate entirely new spread-rate limiting mechanisms, different than the normal-gravity counterpart. These tests are also expected to demonstrate lower flammability limits of solid fuels than any observed in normal gravity. Additionally, this work will attempt to distinguish between influences of buoyantly induced flows and flow of similar intensity that are externally imposed.

**PROGRESS DURING FY 1994:**

This task was completed during fiscal year 1994 with the award of a new flight experiment project entitled, "Combustion of Solid Fuel in Very Low Speed Oxygen Streams," under NRA-93-OLMSA-1, "Microgravity Combustion Science: Research and Flight Experiment Opportunities."

At the transition to the flight program, this task has achieved the following major accomplishments:

1. A complex numerical model has been developed and tested, simulating flame spreading over thin fuels in low-speed concurrent flow. This model includes the full elliptic Navier Stokes equations in the leading edge region of the flame, transitioning to a parabolic, boundary layer formulation downstream to accelerate the computations. The model contains a finite-rate, single-step gas-phase chemical reaction, surface radiation losses, and is able to predict flame spreading behavior, including detailed flame structure over the entire flammable range of concurrent flow velocities at low oxygen concentrations. The model has predicted flammability limits based on blowoff extinction at higher speed flows (similar to observed behavior in normal gravity conditions) and the unique microgravity phenomenon of quenching attributed to the radiative losses at the very low-speed flow velocities. At the time of transition to the flight program, the boundary conditions of the model have been reformulated to permit computations at varying levels of gravitational acceleration.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

2. Based on these computational results, and the related experimental work, a successful proposal for continued development of the theoretical model to include transient behavior and gas-phase radiation processes and the development of a new flight experiment to obtain observations of forced-concurrent flow flame evolution and spreading was submitted to the NRA-93-OLMSA-1 competition.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	1
PhD Students:	1	PhD Degrees:	2

TASK INITIATION: 5/89 EXPIRATION: 2/94

PROJECT IDENTIFICATION: 962-22-05-25

NASA CONTRACT No.: NAG3-1046

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Grayson, G.D., Sacksteder, K.R., Ferkul, P.V., and T'ien, J.S. Flame spreading over a thin solid in low speed concurrent flow: Drop tower experimental results and comparison with theory. *Microgravity Science and Technology*, vol. 7, no. 2, 187-195 (1994).

#### Presentations

Garbinski, G. and T'ien, J.S. "Ignition and extinction of diffusion flames with radiative losses." Presented at the AIAA 32nd Aerospace Sciences Meeting, AIAA-94-0577, 1994.

*Interactions Between Flames on Parallel Solid Surfaces*

PRINCIPAL INVESTIGATOR: Dr. David L. Urban

NYMA, Inc.

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of the proposed research is to study the interactions between flames which are spreading or established over parallel solid surfaces and to determine the relative importance of radiative and convective heat exchange versus the potential reduction of oxidizer due to the presence of the second fuel surface. The results from this work will be used to improve existing models of flame spread over solid surfaces and to improve our understanding of fire spread and growth for practical fuel geometries.

**TASK DESCRIPTION:**

Low gravity testing will be conducted at the NASA LeRC drop towers and on NASA's low-gravity research aircraft. This work will determine the dependence of flame spread over parallel sheets on geometric parameters such as height and width and to use this understanding to select the optimum configuration for more extensive tests. These tests will determine the dependence of spread rate for parallel surfaces on fuel thickness, separation distance, oxidizer flow direction and rate and gas phase radiation. In addition, the controlling factors in the unstable regime at close surface separations will be established. Tests will also be conducted to determine the flame structure and burning rate for flames established over parallel thick fuels with forced flow between the plates. Finally, these results will be combined with numerical/analytical results to determine the importance of radiative feedback, between interacting flames and surfaces, on flammability and spread and burning rates.

**TASK SIGNIFICANCE:**

The interaction between flames on solid surfaces is of considerable practical importance since in most practical heterogeneous combustion systems, the condensed phase is distributed in more than one piece and this spatial distribution can strongly affect burning and/or spread rate. From a fundamental viewpoint, the interaction between parallel surfaces offers opportunities to simplify the boundary conditions, over those for isolated surfaces, as the opposing surfaces impose a plane of symmetry between the two surfaces and if the surfaces are sufficiently large, the radiation shape factors between the surfaces and between the flame and the surfaces approach unity. This geometry also provides interesting and unique opportunities to explore the relative effects of reactant diffusion rates, radiative heat transport and conductive transport in flame spread; these transport mechanisms have been identified as critically important in microgravity.

These phenomena will be more readily studied in nonbuoyant flames as the buoyant transport will be removed, simplifying analysis and increasing the importance of conduction and radiation. In the absence of strong buoyant flows, the interaction between flames on opposing sheets extends to greater separation distances, providing greater opportunities to study the interaction via the greater spatial resolution afforded by microgravity.

These results will be significant to low gravity flame spread research in general as the parallel geometry with its plane of symmetry between the fuel surfaces is in some terms simpler than the traditional case of a single sheet in an infinite oxidizer. The results will be an extension of the existing body of knowledge concerning flame spread and burning rate over flat surfaces. The work might also have practical benefits as radiant preheating is also very significant in fire spread in building fires. Due to the significance of flame interaction in terrestrial fire safety, it is important that flame interactions be studied in low gravity to assess their importance in spacecraft fire safety.

### PROGRESS DURING FY 1994:

Tests were conducted on the KC-135 using the Spacecraft Fire Safety Facility (SF)<sup>2</sup> on its first flight campaign. The tests were very successful, further tests are planned once data analysis has been completed. Samples were burned at one atmosphere in air under flows of 5 and 10 cm/s. Samples were for a variety of separation distances and included single double and triple interacting sheets.

The video tapes from the KC-135 campaign were analyzed to determine spread rate. Instability and oscillating flames were seen at low pressures near the flammability limit.

A large number of 1-g tests were conducted in air at various separation distances and pressures. The flammability limit as a function of pressure and separation has been mapped out for these conditions. At large separations, spread rate is largely independent of pressure until extinction, however at small separations, the spread rate exhibits the change in pressure dependence seen in low gravity.

Over the summer, 170 1 and 0-g tests were conducted at various separation distances and pressures and oxidant concentrations. The low gravity test program expanded the test matrix at 30% oxygen and added points at 21% oxygen. The test program was limited by the current unavailability of gas mixing capabilities in the NASA LeRC 2.2-Second Drop Tower.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94 EXPIRATION: 5/98  
PROJECT IDENTIFICATION: 962-22-05-52  
RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Presentations

Urban, David L. "Interactions between flames spreading over parallel solid sheets of paper in microgravity." A poster presentation was presented at the 25th International Symposium on Combustion.

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*Gasless Combustion Synthesis from Elements Under Microgravity: A Study of Structure-Formation Processes*

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PRINCIPAL INVESTIGATOR: Prof. Arvind Varma

University of Notre Dame

CO-INVESTIGATORS:

Dr. Paul J. McGinn

University of Notre Dame

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**TASK OBJECTIVE:**

The objective is to understand the mechanisms of structure formation during combustion synthesis from elemental powders, in order to find the most promising ways to control the microstructure and properties of advanced materials. Many fundamental questions may be answered by elucidating the reaction mechanism under microgravity conditions, where complicated processes caused by gravity can be ignored.

**TASK DESCRIPTION:**

The reaction systems have been studied previously under normal gravity conditions, and the results have shown that combustion and structure formation mechanisms involve several stages including melting of reactants and products, spreading of the melt, droplet coalescence, diffusion and convection, buoyancy of solid particles and bubbles in the melt, nucleation of solid products, and crystal growth. Most of these processes are affected by gravity. Microgravity experiments will permit the general mechanisms of structure formation to be obtained without the disturbing effects of gravity.

The experimental program includes two approaches: macrocombustion, involving combustion synthesis of pellets pressed from reactant powders, and novel microcombustion experiments where reactions of individual reactant particles are studied. Both approaches will be carried out under normal and microgravity conditions.

**TASK SIGNIFICANCE:**

The research results will be helpful to U.S. industry in its efforts to produce advanced materials with unique properties. They will also establish whether better materials can be produced under microgravity conditions. Both poreless cast and porous foam materials will be developed from the project. These have a significant potential for use as high temperature materials in aerospace and other industrial applications owing to their excellent mechanical, thermal, and chemical properties. The research effort will also help to answer many fundamental questions in the field of combustion synthesis.

**PROGRESS DURING FY 1994:**

Purchase of equipment, system design, and some normal gravity experiments were carried out since the grant was awarded in July 1994. The design of the reaction chamber for the NASA LeRC 2.2-Second Drop Tower was initiated. A computer and data acquisition board were purchased; other equipment including a video recording system, data acquisition, image analysis system, and power supply will be purchased early in FY 95.

Combustion synthesis of the Ni-Al system was carried out. Elemental Ni and Al powders, as well as clad Ni/Al composite powders, were both used as reactants. These powders were then pressed into green pellets with densities about 77% of theoretical, and then ignited in an inert atmosphere. The combustion of elemental powders propagated in a spin mode. In contrast, combustion of clad particle pellets occurred in a rather complex mode which has never been reported. Firstly, the pellets were more difficult to ignite. Secondly, the ignition position was not always at the same end as the ignitor. Ignition sometimes occurred at the opposite end or at some distance away from the ignitor. Thirdly, the combustion wave propagated in a complex mode.

The product microstructures were determined by X-ray diffraction, SEM, and optical microscopy. A structure formation sequence was observed for the clad particles. Namely, intermediate phases were first formed before the

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

microstructure attained the final equilibrium phases. No difference in final equilibrium microstructures was found between the samples from clad and elemental powders.

The reasons for the above phenomena are still being investigated. Further experimental work must be done in order to understand the governing mechanisms.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 3

MS Students: 0

PhD Students: 1

TASK INITIATION: 7/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-22-05-56

RESPONSIBLE CENTER: LeRC

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*Studies of Wind-Aided Flame Spread Over Thin Cellulosic Fuels in Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Indrek S. Wichman

Michigan State University

## CO-INVESTIGATORS:

Prof. Columbo DiBlasi

University of Naples

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TASK OBJECTIVE:

The objective of this research is to develop fundamental theoretical models for wind-aided flame spread over thin solid fuels in low gravity. An important aspect of this study is to develop detailed models for the combustion process in the flame anchoring region, the so-called "Triple-Flame" zone and for the fuel pyrolysis chemistry.

## TASK DESCRIPTION:

The theoretical modeling effort will explore two tracks, one fundamental, the other directly concerned with the wind-aided flame spread calculations. The fundamental part will be detailed examination of the triple flame problem near the leading edge of the spreading flame. The results of this model should provide the heat flux distribution to the supporting fuel surface. The solution techniques employed will be asymptotic and approximate methods combined with numerical methods. The second model to be examined will be wind-aided flame spread calculations. An order-of-magnitude analyses of the governing equations will be performed to identify the correct scaling of the wind-aided flame spread problem, particularly near the flame attachment point. The properly scaled equations will be solved numerically to obtain flame spread rates. The results of this study will be compared against experimental data obtained in low gravity facilities at the NASA Lewis Research Center as a part of other on-going programs.

## TASK SIGNIFICANCE:

Theoretical models for the combustion process occurring at the flame anchoring point will be developed to better understand the triple flame structure. Using this basic model, numerical codes will be developed to predict wind-aided flame spread rates over thin solid fuels.

## PROGRESS DURING FY 1994:

A preliminary version of a monograph (~100 pages) reviewing the current state of knowledge of triple flame structure was prepared by the PI. This monograph identifies the critical areas for future research. Two graduate students have started working on various aspects of the triple flame structure.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 3/94 EXPIRATION: 3/98

PROJECT IDENTIFICATION: 962-22-05-53

RESPONSIBLE CENTER: LeRC

### *High Pressure Droplet Combustion Studies*

PRINCIPAL INVESTIGATOR: Prof. Forman A. Williams

University of California, San Diego

#### CO-INVESTIGATORS:

Prof. M. Kono  
Prof. T. Niioka

University of Tokyo, Japan  
Tohoku University, Japan

#### TASK OBJECTIVE:

The focus of this international cooperative effort is on high-pressure combustion of miscible binary fuel droplets. This is a joint research program pursued by investigators at the University of Tokyo, the University of Tohoku, the University of California, San Diego, and the NASA Lewis Research Center (LeRC). It involves construction of an experimental apparatus in Tokyo and mating of the apparatus to a NASA-LeRC 2.2-Second Drop Tower in Cleveland. Experimental results are to be analyzed jointly by the Tokyo, UCSD, and NASA investigators. The project was initiated in December, 1990, and has now involved three periods of drop-tower testing by Mr. Masato Mikami (U. of Tokyo) at LeRC.

#### TASK DESCRIPTION:

The research accomplished thus far concerns the combustion of individual fiber-supported droplets of mixtures of n-heptane and n-hexadecane, initially about 1 mm in diameter, under free-fall microgravity conditions. Ambient pressures ranged up to 3.0 MPa, extending above the critical pressures of both pure fuels, in room-temperature nitrogen-oxygen atmospheres having oxygen mole fractions,  $X$ , of 0.12 and 0.13.

#### TASK SIGNIFICANCE:

The general purpose is to study near-critical and super-critical combustion of the droplets and to see whether three-stage burning, observed at normal gravity, persists at high pressures in microgravity.

#### PROGRESS DURING FY 1994:

Masato Mikami of the University of Tokyo performed drop tower tests on the burning of droplet arrays of a bicomponent fuel at high pressures. The experimental apparatus was modified to incorporate two video cameras instead of the cine cameras (which necessitated changes to the electronics, camera mounts and the installation of fiber optic transmitters). The gas mixing system, removed during the drop tower construction, was also reassembled for the tests.

After modifications to his experimental apparatus were complete, M. Mikami performed experiments on a two droplet array of two mixtures of heptane/hexadecane (0.80 and 0.90 heptane by volume) at pressures ranging from 1 to 50 atm (above the critical pressures of both fuels). The three staged burning exists for the droplets with in many cases the flames initial separate, then merging during the first stage, then the flames separate during the second stage and merge again during the third stage. Masato returned to Tokyo at the end of September, when he will begin preparing his Ph.D. thesis.

The data taken during August and September was analyzed. It is likely that more data will be required as the success rate was only 50%. This results mainly from the difficulty in igniting the droplets at high pressure.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/89 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-22-05-41

RESPONSIBLE CENTER: LeRC



*High-Pressure Combustion of Binary Fuel Sprays*

PRINCIPAL INVESTIGATOR: Prof. Forman A. Williams

University of California, San Diego

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of the proposed research is to improve understanding of the combustion of sprays of multi-component fuels at elevated pressures, extending from normal atmospheric pressure to pressures above the critical pressure of the fuel. In particular, explanation of the role of previously observed three-stage droplet combustion behavior and microexplosion in spray combustion is sought. The extent to which buoyancy influences the phenomena that occur is to be determined.

**TASK DESCRIPTION:**

The objective is to be achieved by theoretical analyses taking into account concentration profiles of fuel constituents within the liquid, instabilities, droplet interactions, and conditions for achieving the limit of superheat, along with drop-tower experiments employing 1.4 and 2.2-Second Drop Towers. Binary mixtures of heptane and hexadecane will be studied first, using fiber-supported droplets of diameters from 0.8 mm to 1.5 mm in air at pressures from 0.1 to 5 MPa. Later work is planned to involve free droplets and other fuels and possibly microgravity facilities that afford longer test times.

This program is a joint program with several investigators in Japan. The Science and Technology Agency of Japan is supporting research by Dr. Michikata Kono, Professor, Department of Aeronautics, The University of Tokyo, Tokyo, Japan, Dr. Takashi Niioka, Professor, Institute of Fluid Science, Tohoku University, Sendai, Japan, and Dr. Jun'ichi Sato, Senior Researcher, Heat and Fluid Dynamics Department, Research Institute, Ishikawajima-Harima Heavy Industries Co., Tokyo, Japan on "Combustion of a Fuel Droplet in High-Pressure Atmospheres under Microgravity Conditions" and on "Ignition of Fuel Droplets in High Pressure, High-Temperature Environments."

**TASK SIGNIFICANCE:**

The proposed research will improve our understanding of the mechanisms of the combustion of high pressure fuel sprays, such as those found in diesel and gas turbine engines.

**PROGRESS DURING FY 1994:**

Masato Mikami of the University of Tokyo performed drop tower tests on the burning of droplet arrays of a bicomponent fuel at high pressures. The experimental apparatus was modified to incorporate two video cameras instead of the cine cameras (which necessitated changes to the electronics, camera mounts and the installation of fiber optic transmitters). The gas mixing system, removed during the drop tower construction, was also reassembled for the tests.

After modifications to his experimental apparatus were complete, M. Mikami performed experiments on a two droplet array of two mixtures of heptane/hexadecane (0.80 and 0.90 heptane by volume) at pressures ranging from 1 to 50 atm (above the critical pressures of both fuels). The three staged burning exists for the droplets with in many cases the flames initial separate, then merging during the first stage, then the flames separate during the second stage and merge again during the third stage. Masato returned to Tokyo at the end of September, when he will begin preparing his Ph.D. Thesis.

The data taken during August and September was analyzed. It is likely that more data will be required since the success rate was only 50%. This results mainly from the difficulty in igniting the droplets at high pressure.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/94    EXPIRATION: 11/98

PROJECT IDENTIFICATION: 962-22-00

RESPONSIBLE CENTER: LeRC

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*Laser Diagnostics for Fundamental Microgravity Droplet Combustion Studies*

PRINCIPAL INVESTIGATOR: Dr. Michael Winter

United Technologies Research Center

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

This research program seeks to investigate the range of applicability of advanced diagnostics to droplet combustion under microgravity conditions while generating fundamental information relevant to other ongoing NASA programs. Using a progressive approach, advanced laser diagnostics will progress from laboratory, to flight experiments, and ultimately to drop tower facilities. In this way, these diagnostics can be developed, while providing fundamental data on droplet transport and combustion phenomena. Through consultation with NASA personnel, and other researchers in the fields of microgravity droplet transport and combustion, priorities have been established with respect to measurement parameters. The prioritized measurement parameters are:

1. Flame front position.
2. Relative gas-phase flow around droplets.
3. Droplet surface transport and internal flow.
4. Liquid-phase thermometry.

**TASK DESCRIPTION:**

An integrated diagnostic unit, capable of performing these measurements has been assembled. All of the major components such as lasers, detectors and data systems, are currently allocated to this effort. A self-contained, miniature, nitrogen-pumped dye laser system and two-dimensional intensified CCD imaging system will form the core of the unit.

The testing program is proceeding with ever more challenging environments proceeding from the 1g laboratory, to aircraft, and finally, to drop tower facilities. The diagnostic approaches and instrumentation are being verified in a laboratory setting under Task II. Experiments with the intent of reducing the risks associated with high impact and droplet drift are being conducted on the aircraft, with fiber suspended droplets under Task III. Initial measurements are being performed on use of PLIF measurements to describe flame front position. Gas-phase flow dynamics will be determined by velocity measurements using particle image velocimetry. Measurement of droplet surface and internal transport will be attempted later in the program. The applicability of liquid phase thermometry in a burning droplet is being pursued at NASA LeRC and frequent consultation toward that effort is being provided by UTRC.

**TASK SIGNIFICANCE:**

Optical diagnostics offer non-intrusive measurements of either scalars or vector quantities in multipoint measurements. Experimental droplet combustion data critically tests the combustion theory and provides input for modelling spray combustion used in combustors.

**PROGRESS DURING FY 1994:****Hardware:**

An apparatus for performing laser-induced fluorescence measurements of fiber supported burning fuel droplets aboard the NASA Learjet and KC-135 aircraft facilities was designed and built primarily in 1993 with testing and use in 1994. The rig houses a self-contained miniature nitrogen-pumped dye laser system and two-dimensional intensified

CCD and unintensified CCD imaging systems. Two time code generators, two VCR's, a timing system, and MacIntosh IICI computer with image digitization hardware form the data collection/storage system. Droplet deployment, ignition and synchronization to the timing electronics is accomplished via a unique system of radio controlled model airplane controls.

### Experiments:

During the week of December 13, 1993, a series of tests were performed on the NASA Lewis Learjet aircraft. Two experiments were attempted during these flights, planar laser-induced fluorescence (PLIF) and particle image velocimetry (PIV). The PLIF experiments were performed using sodium as the fluorescing species originating from sodium seeded methanol droplets. These images provide information on the flame front position for comparison with theoretical calculations. The PIV experiments utilized 0.9  $\mu$ m silica particles entrained into the flow ~ 15 seconds prior to ignition. These experiments were less successful as upon ignition, the heat release and corresponding gas expansion propels the PIV seeds out of the measurement volume.

During the week of April 11, 1994, aircraft experiments were again conducted. Tests were directed at the following:

1. Ignition vs. ignitor stand-off distance for methanol.
2. Radical chemiluminescence with simultaneous visible luminosity images.
3. PLIF images from burning methanol droplets with simultaneous visible luminosity in NaCl seeded methanol droplets.
4. PLIF images from burning ethanol droplets with simultaneous visible luminosity in NaCl seeded ethanol.

### Results:

Ignition standoff data for the methanol indicated that the ignitor wire needed to be as close as physically possible to achieve reliable ignition. In contrast, the position of the ignitor needed to be several diameters away from the droplet surface to achieve reliable ignition in ethanol.

Successful radical chemiluminescence, PLIF and visible flame luminosity images were obtained. Initial analysis indicates that the broadband visible flame luminosity and radical chemiluminescence agree well in mapping the flame front position. Comparison between the broadband visible flame luminosity and simultaneous PLIF images of NaCl seeded burning methanol droplets indicates that the visible luminosity is not a reliable marker of the flame front position, being dominated by Na chemiluminescence for droplets seeded with NaCl.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/92 EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-22-05-44

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Presentations

Winter, M. "Laser diagnostics for microgravity droplet studies." AIAA 32nd Aerospace Science Meeting and Exhibit, Reno, NV. AIAA Paper 94-0431, January 10-13, 1994.

*Combustion of a Polymer (PMMA) Sphere in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Jiann C. Yang

National Institute of Standards and Technology (NIST)

CO-INVESTIGATORS:

Dr. Anthony Hamins

National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE:**

The Objective is to investigate the combustion characteristics of spherical PMMA particles in reduced gravity.

**TASK DESCRIPTION:**

Experiments will be conducted to study both supported and unsupported PMMA particles burning in a controlled environment under microgravity conditions. The test conditions will be room temperature and pressure (25 °C and 1 atm). The ambient oxygen concentration will range from 19% to 70% and the particle size will be 3, 5 and 7 mm. Different ignition methods will also be tested. The diagnostic system will consist of a CCD video camera and a high speed movie camera. The video and film images will be analyzed to provide information on burning history, flame location, soot formation, burning rate, and other combustion characteristics.

**TASK SIGNIFICANCE:**

This experiment is the first attempt to study the combustion of a solid fuel sphere under microgravity conditions. The one-dimensional configuration will simplify the data interpretation and the test results will provide some fundamental information to solid fuel combustion science.

**PROGRESS DURING FY 1994:**

Preliminary ignition experiments of PMMA spheres were conducted with different devices. The spheres of 5 mm in diameter were easily ignited with a small propane torch. However, ignition of the sphere (without preheating) could not be achieved when a spark ignitor (with an on-time of more than 2 secs) was used. Ignition of solid PMMA spheres was then further tested. Spark ignition of a PMMA particle coated with a thin layer of ethanol was unsuccessful. Neither was with a preheated particle. However, ignition was always achieved using a small torch or a match. The PI concluded that a micro-torch might be the appropriate means to ignite particles. Therefore, a micro-torch is being designed for ignition. The system is very similar to a cigarette ignitor using liquid butane.

The fabrication method of PMMA spheres with embedded thermocouples was devised and planned. PMMA powder will be dissolved in a solvent and an initiator (benzoyl peroxide) will then be added to the solution. The polymer solution will be cured in an oven into a spherical mold and a thermocouple inserted into the solution. Some technical problems still remain to be solved, e.g. how to avoid bubble formation in the PMMA sphere. A technique is also being tested to accurately embed a thermocouple junction at desired location inside a PMMA particle. The first step is to heat the thermocouple by passing a electric current through it. Then the heated thermocouple is lowered into a PMMA particle by using a linear positioning stage.

Some experimental equipment were consolidated. A LO-CAM (model 50) was located in-house at NIST. A scanner capable of digitizing 16 mm films was also acquired. Imaging software such as SigmaScan/Image is needed.

Dr. J. C. Yang (PI) and Dr. A. Hamins (Co-I) visited NASA Lewis Research Center and toured the microgravity facilities. They obtained first-hand information on rig construction, test procedures and other related issues through a series of discussions with people from the Space Experiment Division.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94    EXPIRATION: 4/98

PROJECT IDENTIFICATION: 962-22-05-63

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

### *Study of Two-Phase Flow and Heat Transfer in Reduced Gravities*

PRINCIPAL INVESTIGATOR: Dr. Davood Abdollahian

S. Levy, Inc.

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective is to conduct two-phase flow instability studies in vertical upflow and downflow, in both a normal gravity environment and in low gravity aboard an aircraft, to ascertain the effect of gravity on instability and boiling mechanisms.

#### TASK DESCRIPTION:

The approach is to design and build a recirculating flow boiling loop which would be used aboard an aircraft to test different two-phase flow instability phenomena and measure the conditions at which critical heat flux occurs. Specifically, the instability phenomena to be examined are the following: nucleation instability, flow pattern instability, excursive instability, oscillatory instability, and density wave instability. Testing would consist of examining the flow stability and critical heat flux in normal-gravity vertical upflow and downflow as well as in low gravity aboard an aircraft.

#### TASK SIGNIFICANCE:

Two-phase instabilities have been responsible for a multitude of fatal and costly accidents on Earth in the oil, electric power and nuclear industries. In addition, by understanding critical heat flux better, it may be possible to improve the cooling of microcircuitry used in the electronic industry thus improving the capability of that circuitry.

#### PROGRESS DURING FY 1994:

Design of the electrical and flow systems and the test sections was completed and approved by the NASA COTR. Construction of the flow loop and test sections was initiated and completed. The data acquisition and control system software was written and is being debugged. A Lab Windows package is being incorporated.

Two Learjet equipment racks, a high-speed camera body and mount, and a mockup of a SAMS accelerometer were furnished as government furnished property. The flow loop, camera, data acquisition and control system, and accelerometer are being incorporated into the racks.

The test sections are mounted to a support structure other than the Learjet racks for normal gravity testing. This permits vertical upflow and downflow conditions to be examined. Shakedown testing of the flow loop has commenced.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 3  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 3/93 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-24-05-50

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Presentations**

Abdollahian, D. and Barez, F. "Study of two-phase flow and heat transfer in reduced gravities." Presented at Second Microgravity Fluids Conference in Cleveland, OH. June 21-23, 1994.



*Colloids & Nucleation*

PRINCIPAL INVESTIGATOR: Prof. Bruce J. Ackerson

Oklahoma State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Direct observations will be made of the nucleation and early growth of crystals comprised of polymethylmethacrylate (PMMA) "hard" colloidal spheres in an index matching solvent. Colloidal samples will be prepared in a particle volume fraction range which exhibits crystals of colloidal particles at equilibrium. These samples will be shear melted and the subsequent appearance of crystals monitored by direct observations/video camera recording at the first order Bragg scattering angle.

**TASK DESCRIPTION:**

1. Apply classical nucleation theory in the limit applicable to hard sphere interactions to compare against data. Modern analytic (density functional) and simulation results will be compared with data as appropriate.
2. Establish the practical limiting density supersaturation that can be studied without interference due to sedimentation induced convection.
3. Conduct an exploratory investigation of the density or order parameter fluctuations leading to a critical nucleus, and if possible, characterize the size and time dependent characteristics of these fluctuations.

**TASK SIGNIFICANCE:**

The experimental observations of nucleation and early growth of crystals comprised of "hard" colloidal spheres will provide a critical test of classical nucleation theory.

**PROGRESS DURING FY 1994:**

Work has been initiated, but to date no substantial progress has been made.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-78

RESPONSIBLE CENTER: LeRC

### *Stability Limits and Dynamics of Nonaxisymmetric Liquid Bridges*

PRINCIPAL INVESTIGATOR: Prof. Iwan D. Alexander

University of Alabama, Huntsville

CO-INVESTIGATORS:

Dr. J.M. Perales

Dr. J. Meseguer

Universidad Politecnica de Madrid

Universidad Politecnica de Madrid

#### TASK OBJECTIVE:

The objectives of the proposed work are:

1. To determine the stability limits of Nonaxisymmetric liquid bridges held between non-coaxially aligned disks.
2. To examine the dynamics of Nonaxisymmetric bridge configurations and Nonaxisymmetric oscillations of initially axisymmetric bridges.
3. To experimentally investigate the vibration sensitivity of liquid bridges under terrestrial and low gravity conditions.

#### TASK DESCRIPTION:

The program is to have simultaneous experimental and theoretical efforts. Experimentally, normal-gravity tests using the Plateau method will be conducted to study the equilibrium shapes and stability limits of various orientations of the liquid bridge, and to study the sensitivity of liquid bridges to axial and lateral vibration.

A numerical model will be developed using Picard iterative procedure to study the dynamics of Nonaxisymmetric bridges subject to g-jitter and the vibration sensitivity of liquid bridges.

#### TASK SIGNIFICANCE:

Liquid bridge stability is an important factor in determining the stability of molten zones associated with floating zone crystal growth experiments. Such understanding can help better define the vibration isolation requirements for in-space processing experiments, and can greatly enhance the chances of obtaining new and better quality semi-conductor crystals.

#### PROGRESS DURING FY 1994:

In FY94, the stability limit of bridges held between coaxial disks was experimentally studied. The transition from symmetric to asymmetric configuration was observed, and numerical analyses were conducted to investigate the effects of gravity orientation on the maximum and minimum volume stability limits. A novel technique for measuring the force applied to the support disks has been developed and applied to dynamic lateral shearing and squeezing experiments. A neutral buoyancy tank has been constructed to allow for vertical and lateral low frequency vibration of the support disks, and to allow for independent rotation of the two support disks. In addition, a theoretical investigation has been conducted to study the effects of vibration on the stability limit of liquid bridges. The study shows that the bridge may be stabilized or de-stabilized relative to the static stability margin depending on the nature of the axial vibration.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-60

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Meseguer, J. , Gonzalez, M.A. and Alexander, J.I.D. Dynamic stability of long axisymmetric bridges. Microgravity Science and Technology, VII, 234-242 (1994).

*Investigations of Multiple-Layer Convection*

PRINCIPAL INVESTIGATOR: Prof. C. D. Andereck

Ohio State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The convection patterns that develop in two-layer fluid systems are to be studied. The principal objectives are:

1. To test the predictions of theoretical investigations.
2. To study the interaction of the pattern in one layer with the pattern in the other layer.
3. To determine the role of the geometry of the test cell on the patterns that emerge.
4. To determine the impact of surfactants at the fluid interface on the interface profile and the patterns that form.

**TASK DESCRIPTION:**

An experimental investigation of natural, buoyancy driven, convection in a system consisting of two superposed layers of immiscible fluids will be performed. Narrow, pseudo-one-dimensional, test cells will be constructed with insulating side walls and conducting top and bottom walls. Temperature control of the horizontal surfaces will be provided initially by circulating known temperature water next to these surfaces. Shadowgraph visualization techniques, which are sensitive to index of refraction variations resulting from temperature gradients in the fluids, will be used to determine the flow patterns. This allows for observations of the basic dynamics of competing instabilities in the two layers. A direct comparison will then be possible between existing general theories of pattern competition, and experimental results for a specific system. This will also provide a test for direct numerical simulations of two fluid layer systems. Further work to be performed includes studying the deformation of the interface as a function of control parameter and with the introduction of surfactants. Finally, the geometry of the cell will be varied. To introduce periodic boundary conditions in one dimension we will construct an annular cell. Another variation will be to construct a cell with large extent in both horizontal dimensions, thereby freeing the system to choose more complex competing patterns.

**TASK SIGNIFICANCE:**

Multiple-layer convection is of interest in geophysical, astrophysical and industrial settings. It is also of importance as a fundamental pattern forming nonequilibrium system. These experiments will provide a quantitative test of our theoretical understanding of this fluid dynamical system. Results on the pattern formation processes may also be directly relevant, in thin layer limits, to understanding and improving film coating processes.

**PROGRESS DURING FY 1994:**

In the startup phase of the grant we have built a first version of the test cell and have used shadowgraph imaging to view the patterns. The cell is a narrow rectangular box, with the top and bottom walls of copper and the side walls of a single block of machined and polished plexiglass. The shadowgraph system consists of a HeNe laser with a spatial filter, beam expander and large collimating lens, which produces a reasonably intense parallel beam that passes through the side of the cell. This is followed on the other side with a screen and video camera for image capture and analysis. This has been tested for a single layer of silicone oil. We are now testing various combinations of silicone oils, mineral oils, and other liquids to find those that provide optimum matches for the two-layer experiments. A particular problem is matching the viscosity and thermal conductivity in such a way that the two layers will become unstable to their respective patterns simultaneously. A further criterion is that the fluids be immiscible, at least roughly so over the time scales of the experiments.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Our plans for the remainder of this year will include performing as detailed a study as possible of the interaction of the patterns for different layer depth ratios, particularly those that lead to resonant interactions, and building two other test cells. The first will be an improved version of the present rectangular geometry, with better temperature control and attention to the sidewall conditions. A second cell will be annular in shape, to eliminate the end boundaries of the cell and allow for patterns that are free of any pinning, driving or forcing effects of the end walls.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-79

RESPONSIBLE CENTER: LeRC

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### *Electrokinetic Transport of Heterogeneous Particles in Suspensions*

PRINCIPAL INVESTIGATOR: Prof. John L. Anderson

Carnegie Mellon University

CO-INVESTIGATORS:

S. Garoff

Carnegie Mellon University

#### TASK OBJECTIVE:

There are three objectives to our research program. The first is to complete a hydrodynamic theory for the motion of slender colloidal particles in electric fields. The other two objectives involve experimental measurements. The first of these is to design and develop a microelectrophoresis apparatus and video imaging system that would enable us to follow the kinematics of colloidal doublets in electric fields. The second experimental objective is a study of the rotational rates of doublets of latex particles in solutions at different ionic strength.

#### TASK DESCRIPTION:

1. The electrophoresis of nonuniformly charged slender particles, such as cylinders and prolate spheroids, is analyzed in terms of existing models for the hydrodynamics of such particles. The basic idea is to apply the Lorentz reciprocal theorem of Stokes flow with the concept of electroosmotic "slip velocity" on the surface of the particle to obtain explicit algebraic expressions or quadratures for the translational and rotational velocities of the particle of arbitrary contour shape (straight, curved, helical, etc.). This theory can be directly extended to model the electrophoresis of uniformly charged slender particles in nonuniform electric fields.

2. The microelectrophoresis apparatus must permit observation of particle motion in a constant, one dimensional electric field at field strengths ranging from 0.1 to 50 V/cm. The optical quality of the cell is important. In addition, a method for recording and analyzing position/orientation versus time for single particles and aggregates must be developed. Temperature control to better than  $\pm 1^\circ\text{C}$  is desired to avoid complications of convection; however, in analyzing the rotational motion natural and forced convection resulting from the electrical field is not too much of a problem. In addition to developing this apparatus, the images must be analyzed in a way to interpret the data in terms of the existing hydrodynamic models for electrophoretic rotation.

3. Colloidal doublets formed by coagulating two latex particles of different zeta potential (i.e., different surface charge) rotate when an electric field is applied. Two theoretical models, differing only in the physical picture of the doublet, are available for the angular velocity of the doublet as a function of sphere sizes and zeta potentials. We are collecting data at different solution conditions (pH, ionic strength) for comparison with these two models.

#### TASK SIGNIFICANCE:

1. Many colloidal systems are not composed of spherical, uniformly charged particles. For example, clays are disks with edges that differ from the faces, and most inorganic colloids have different crystal planes exposed at the surface thereby generating a "patchiness" to the charge. Aggregates of particles also create heterogeneous supraparticles that could result in chains with a distribution of zeta potential along their length. Our theoretical work on electrophoresis of charged slender particles expands the range of understanding heterogeneous systems.

2. The now classical DLVO theory of the energetics between two colloidal particles in aqueous media predicts two states of adhesion between two spheres. The first is a "primary minimum" in the potential energy which occurs when the two spheres essentially touch. The second state of adhesion is in the "secondary minimum" where electrostatic repulsion balances attraction caused by London dispersion forces; the secondary minimum is characterized by gap distances of order 4-8 Debye screening lengths. From hydrodynamics we expect that a doublet in the primary minimum would rotate like a rigid body in an electric field, with the more positive sphere rotating toward the field direction, while in the case of a secondary minimum the apparent doublet rotation occurs with each

sphere rotating at a different angular velocity to keep the hydrodynamic torque on it equal to zero. The difference in rotation rate predicted by theory for these two cases is a factor of 3. We believe that electrophoretic rotation is a tool to probe the state of adhesion of colloidal aggregates.

### PROGRESS DURING FY 1994:

1. The theoretical model for electrophoresis of slender particles has been developed and published. Some remarkable predictions are made by the theory in the transport of long straight particles in nonuniform electric fields as would be realized at the entrance of pores in a membrane. These predictions form the basis of an experimental program that we will initiate soon.

2. The microelectrophoresis apparatus has been developed with video imaging capabilities. A moment analysis has been developed to interpret orientation versus time data to obtain the fundamental parameters (electrophoretic rotation, Brownian diffusion coefficient) needed to characterize the heterogeneous colloidal doublets of latex that are being studied. Data are currently being collected and analyzed.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-24-08-09

NASA CONTRACT No.: NAG8-964

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### **Presentations**

Anderson, J.L. "Electrophoresis of nonuniformly charged chains." Symposium on Electrophoresis, American Chemical Society Meeting, Denver, CO, 1993.

Anderson, J.L. "Electrophoresis of heterogeneous aggregates of colloidal particles." AIAA 32nd Aerospace Sciences Meeting, Reno, NV, January 10-13, 1994.

Anderson, J.L. "Electrophoresis of heterogeneous colloids: disk and long chains." 8th International Conference on Surface and Colloid Sciences, Adelaide, South Australia, February 13-18, 1994.

Anderson, J.L. "Electrokinetic transport of heterogeneous particles in suspension." NASA Microgravity Fluid Physics Conference, Cleveland, OH, June 12-13, 1994.

Anderson, J.L. "Rotation of colloidal particles by electrophoresis." Seminar given at van't Hoff Laboratory, Kruyt Symposium, Utrecht, The Netherlands, October, 1994.

*Experimental Study of Liquid Jet Impingement in Microgravity: The Hydraulic Jump*

PRINCIPAL INVESTIGATOR: Prof. C. T. Avedisian

Cornell University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The primary objective of this task is to investigate the experimental study of liquid jet impingement in microgravity. The proposed experimental study is aimed at understanding the effects of gravity on a hydraulic jump formed during impingement of a liquid jet on a target plate.

**TASK DESCRIPTION:**

This study is important in applications which involve cooling of a heated surface by a relatively colder liquid jet since the heat transfer rate is strongly affected by the velocity (and hence the thickness) of the liquid film spreading on the surface. The principal investigator plans to examine the effect of gravity on the location of the hydraulic jump created by an impinging water jet onto a flat surface.

**TASK SIGNIFICANCE:**

This experimental study will test the validity of the theoretical predictions, and provide a first step towards more detailed studies of heat transfer and phase change processes associated with jet impingement cooling of heated surfaces in microgravity environments.

**PROGRESS DURING FY 1994:**

The research to study the fluid mechanics of a circular hydraulic jump (CHJ) formally began on July 1, 1994. The first task was to make final arrangements for personnel. The original proposal called for graduate student participation. However, it was later judged that the short duration of the project would be better served by participation of a more experienced person. Accordingly, a post doctoral associate was hired effective July 1, 1994.

The work underway is centering around designing a chamber to create a laminar CHJ. The apparatus will include a nozzle to create the jet plate on which the jet will impinge, circulation pump, fluid containment, and photographic. Perhaps the most critical aspect of the design will be for the fluid jet. It can be created by either a sharp-edged orifice arrangement or by fluid emerging from a tube. The photographic arrangements that is currently being set up is based on 35 mm photography. Various lighting arrangements will be examined to yield the best contrast and image clarity.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-24-00-34

RESPONSIBLE CENTER: LeRC



*Studies on the Response of Emulsions to Externally-Imposed Electric and Velocity Fields:  
Electrohydrodynamic Deformation and Interaction of a Pair of Drops*

PRINCIPAL INVESTIGATOR: Prof. James C. Baygents

The University of Arizona

CO-INVESTIGATORS:

H. Stone

Harvard University

**TASK OBJECTIVE:**

The objective of this study of multiphase electrohydrodynamic flows is to determine the electric field-induced microstructural response of two-phase systems, as well as to develop an improved understanding of the physicochemical and transport process common to a variety of low-gravity flows; we examine the motion, deformation, and interaction of pairs of viscous drops owing to an applied electric field.

**TASK DESCRIPTION:**

The research is a comprehensive numerical and theoretical study of several phenomena common to multiphase flows where electrical fields are used to manipulate the microstructure in the absence of any buoyancy-induced fluid motions. This class of problems appears in typical materials handling, physicochemical processes (e.g., emulsion breaking and drop coalescence) and bioseparations processes (e.g., aqueous two-phase partitioning).

This investigation is designed to use numerical solutions to develop an improved quantitative understanding of the effect of electric fields on typical two-drop (pair) interactions that lead toward coalescence. Cases will be studied where the potential distribution is influenced by conduction processes in the limit where viscous effects dominate the hydrodynamics. Both the effects of uniform and simple nonuniform (imposed) fields will be studied. The "leaky" dielectric model and the Stokes equations will be employed to describe the constitutive behavior of the dielectric media. The numerical calculations will use established integral equation methods for solving this class of time-dependent free boundary problems.

**TASK SIGNIFICANCE:**

The systematic study will lead to a quantitative description and understanding of electrically-driven low-gravity fluid motion, particularly as these flows pertain to the manipulation and positioning of drops and bubbles with electric fields. Such flows are significant for several reasons. First, in the absence of the usual gravitational forces, electric fields can be used to effect phase separation processes that are controlled by drop coalescence (e.g. emulsion breaking). Second, electric fields have long been envisioned as a tool for containerless processing of materials, inasmuch as the fields can be used to manipulate fluid interfaces, such as those of a drop surface, and as a means to levitate fluid globules against weak gravitational fields.

**PROGRESS DURING FY 1994:**

We have successfully implemented and tested numerical solutions to two problems. The first is the axisymmetric electrohydrodynamic deformation and interaction of equal-sized drops subject to the imposition of a uniform electric field. A summary of this work was submitted to the Proceedings of the Second Microgravity Fluid Physics Conference, held in Cleveland, OH, July 1994. A complete exposition of the work is to be submitted for publication at the end of CY94.

The second problem solved is the electrohydrodynamic deformation and translation of a single drop in a nonuniform electric field. Here numerical results have compared favorably to limiting analytical cases for the drop translation, which were worked out as part of this project to provide benchmarks for the numerical studies.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	1
PhD Students:	1	PhD Degrees:	0

TASK INITIATION: 3/93    EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-24-08-13

NASA CONTRACT No.: NAG8-948

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Presentations

Baygents, J.C. "Electrohydrodynamic deformation and interaction of a pair of emulsion drops." Second Microgravity Fluid Physics Conference, NASA Lewis Research Center, Cleveland, OH, June 21-23, 1994.

*Marangoni Effects in Boiling of Binary Fluid Mixtures Under Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Van P. Carey

University of California, Berkeley

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Recent efforts to more fully optimize performance of power, refrigeration and thermal control systems have lead some developers to consider the use of binary working fluids. Vaporization of the working fluid is often a critical element in the performance of such systems. However, transport phenomena associated with vaporization of binary liquid mixtures is still not well understood. The main objective of this project is to separate and explore the roles of buoyancy and Marangoni effects in binary liquid mixture boiling under simulated low gravity conditions. The research will also aim to find coolant mixtures that are particularly well suited for use in a variety of applications including spacecraft thermal control.

**TASK DESCRIPTION:**

This project consist of a series of experimental studies of nucleate boiling of a variety of binary mixtures. The experiments will include both 1-g and low-g data of boiling curve and critical heat flux. Testing the boiling performance of the heating surface in different orientations will establish whether the presence of Marangoni effects reduces the sensitivity of the system to the direction of the gravity vector. The least sensitive mixtures will then become the focus of tests under low gravity conditions (via NASA LeRC drop towers or low-g aircraft).

**TASK SIGNIFICANCE:**

The experimental database for boiling of binary fluid mixtures will help to clarify the individual contributions of gravity and Marangoni effects on the heat transfer from the surface and the critical heat flux transition. It will also help to develop a capability to model these effects with reasonable accuracy to improve the designs of thermal systems.

**PROGRESS DURING FY 1994:**

During this reporting period, the following have been accomplished:

1. A Learjet rack was received from NASA LeRC. The rack is on loan to UC Berkely to be used as the support frame for the experiment.
2. Component assembly is currently in process.
3. Started system design and installationinto the support frame.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-00-81

RESPONSIBLE CENTER: LeRC

*Marangoni Instability Induced Convection in Evaporating Liquid Droplets*

PRINCIPAL INVESTIGATOR: Dr. An-Ti Chai

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

Prof. V.R. Arpaci

University of Michigan

**TASK OBJECTIVE:**

The objectives of the proposed effort are: (1) to study and to characterize the Marangoni instability phenomena in the near ideal configuration of an evaporating droplet in microgravity, and (2) to establish the effect of the induced convection on the droplet evaporation rate.

**TASK DESCRIPTION:**

Specifically, the purpose is to study the Marangoni instability and thermocapillary convections in an evaporating liquid drop in the Fluids Experiment System (FES) developed for flight on Space Shuttle missions. When a liquid drop undergoes evaporation, its surface temperature decreases. If the droplet is free floating in a microgravity environment, the heat transfer process inside the droplet is "conduction controlled." As the process continues, a radial temperature gradient builds up at the free surface until the critical Marangoni number is exceeded. Then the onset of instability induces thermocapillary convective flows that, in turn, speed up the evaporation. The convective flow will subside when the interior of the droplet reaches a certain equilibrium temperature, and the process will return to the "diffusion controlled" mode.

**TASK SIGNIFICANCE:**

When the proposed experiments are successfully completed, the unique data set, which will be collected under near ideal conditions, should by itself constitute a significant contribution to our knowledge of droplet evaporation. The significance and influence of the proposed project go far beyond any unique experimental data set. With the anticipated data set, it will be possible to quantitatively evaluate the well known Maxwell's theory of droplet evaporation. Additionally, it will be possible to establish that thermocapillary instability induced convection inside a droplet can affect its evaporation process. It is our intention to use this information to pursue the development of a complete and comprehensive theory of droplet evaporation.

The value of a more comprehensive and complete theory cannot be overstated. Having a solid theoretical basis, numerical computation can be devised along with skillful scaling techniques to explore cases where direct experimentation is not feasible. The influence of the proposed work is expected to be far reaching in this regard.

**PROGRESS DURING FY 1994:**

Most feasibility issues have been demonstrated experimentally. An analytical and numerical scheme has been applied to model the quasi-static thin shell configuration. Qualitative agreement has been obtained between this model and dimensional analyses.

Our recent analytical effort was to pursue the transient nature of the Marangoni instability problem encountered in an evaporating droplet. As a result, we formally introduced a new fundamental dimensionless number,

$$\Pi_c \sim Ma/(1+Pr^{-1})$$

for thermocapillary driven flows. Here Ma and Pr are respectively the usual Marangoni and Prandtl numbers. The significance of this number for past Marangoni instabilities is demonstrated in terms of a projection method involving the Godunov discretization for convective terms. Particular effort has been made to compare results from the new approach with data available in the literature. However, direct simulation of space-wise distributed problems requires considerable computation time at higher transition. We have begun the exploration of the theory of nonlinear dynamic systems (chaos) which appears to be an appropriate and effective approach.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 962-24-00-65

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Journals**

Arpaci, V.S., Kou, S.H., and Chai, A.T. Chaotics of thermocapillary driven flow past Marangoni instability. Two Fluid Flows-With or Without Phase Change, AMD-vol.184. (1994).

**Proceedings**

Arpaci, V.S., Evren-Selamet, E., and Chai, A.T. "Thermocapillary driven flows past Marangoni instability." AIAA 32nd Aerospace Sciences Meeting. Reno, NV. January 21-23, 1994.

Chai, A.T. and Arpaci, V.S. "A realistic model of evaporation for a liquid droplet." Proceedings of the Second Microgravity Fluid Physics Conference. Cleveland, OH. June 21-23, 1994.

*Rewetting of Monogroove Heat Pipe in Space Station Radiators*

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**PRINCIPAL INVESTIGATOR:** Prof. S. H. ChanUniversity of Wisconsin, Milwaukee

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**CO-INVESTIGATORS:**No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The general objective of the program is to investigate the physics of rewetting of heated, grooved flat plates in a microgravity environment.

Specific objectives of the program are to add to the fundamental understanding of the physics of rewetting/dryout fronts; to better quantify the conditions under which rewetting occurs; and to obtain more accurate predictions of the rewetting velocity for a range of parameters.

**TASK DESCRIPTION:**

The program is composed of both experimental and theoretical investigations. For the experimental investigation a grooved flat plate apparatus will be designed and constructed. Liquid will be introduced on one side of the plate to flow along the grooved surface; the other side of the plate will be electrically heated. This apparatus will be operated both on the ground and aboard a microgravity aircraft.

Data from these experiments will consist of temperature measurements in the liquid film and the heated plate. Advancing wetting front (rewetting), receding wetting front (dryout), and stationary wetting front cases will be examined.

The theoretical analysis will be made by applying experimental data on rewetting velocity and temperatures to analytical processes developed for predicting rewetting velocities on overheated nuclear fuel rods.

**TASK SIGNIFICANCE:**

Understanding the dryout and rewetting characteristics of grooved surfaces is important in predicting behavior of monogroove heat pipe designs proposed for the space station.

**PROGRESS DURING FY 1994:**

Several aspects of experimental apparatus have been modified and improved and laboratory testing has also been undertaken. They are as follows:

1. A new liquid delivery system has been designed to eliminate reliance on gravity to drive flow to the test section. The delivery system was successfully tested to show that the rewetting speed is independent of the liquid supply velocity in both plate orientations, the grooved surface upward-facing and downward-facing positions.
2. A video recording system capable of recording and playing back high resolution pictures frame-by-frame was installed to measure rewetting front locations.
3. A lighting mechanism was installed to synchronize the computer data acquisition time with the video time.
4. The surface temperature measured by two spring-loaded traversing thermocouple probes was tested and found in agreement with the embedded thermocouple.
5. Rewetting experiments were conducted with the plates preheated to various initial temperatures and with plates in the face up and the face down orientations. The heating was provided by a new heater (with a new controller) installed at the end of the plate.

The following theoretical works have also been undertaken:

1. Hydrodynamically controlled and conduction controlled rewetting models were proposed respectively for the plate temperature lower and higher than the wet front temperature.
2. Numerically exact solutions for the rewetting of a plate heated from one end was obtained for various conditions.
3. Approximate analytical solutions were also obtained for the prediction of the wetting speed on finite, initially-isothermal plates subject to heating from the end of the plate. The solution was found in good agreement with the numerical solution.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-54

NASA CONTRACT No.: NAG-1381

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### **Presentations**

Chan, S.H. "Experimental and theoretical studies of rewetting of unheated/heated grooved plates." Presented at and to appear in the proceedings of the Second Microgravity Fluid Physics Conference, 21-23 June 1994.

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*Marangoni and Double-Diffusive Convection in a Fluid Layer Under Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Chuan F. Chen

University of Arizona

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CO-INVESTIGATORS:

C. Chan

University of Arizona

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**TASK OBJECTIVE:**

To study the onset and the subsequent convection in a fluid layer subjected to Marangoni and double-diffusive instabilities, including the effects of cross diffusion and gravity modulation.

**TASK DESCRIPTION:**

A coordinated research effort in ground-based experiments, stability analysis, numerical simulation, and a design sensitivity study is to be conducted. Experimentally, the effect of gravity modulation on the onset and subsequent convection in singly and doubly diffusive layers will be studied with improved experimental techniques. Theoretically, stability analyses of Marangoni double-diffusive instability will be conducted, and a numerical simulation using boundary element method will be carried out to examine the interaction of finger convection with Marangoni convection, and to test the design sensitivity to optimize the flight experiment.

**TASK SIGNIFICANCE:**

The results of this study will enable the design of an apparatus for crystal growing and for casting in a space environment. In addition it will offer an improved understanding of materials processing, which will enhance the chances of obtaining new and better quality crystals for the semi-conductor industry and for the bio-medical industry. This study can also provide an improved understanding of the interaction of surface tension forces and double diffusive buoyancy forces which would lead to the improvement of manufacturing processes on Earth.

**PROGRESS DURING FY 1994:**

An experimental and theoretical investigation of the interactions between Marangoni and double-diffusive instabilities has been completed in FY94. The experiments were conducted in a rectangular tank with dimensions of 25 cm in length, 13 cm in width, and 5 cm in height. A stable solute (NaCl) stratification was first established in the tank, and then a vertical temperature gradient was imposed. A series of stability experiments was conducted for a layer with an initial top concentration of 2 wt% and different concentration gradients. The stability map shows that in the rigid-free case, the early Marangoni instability at the top region reduces significantly the critical Rayleigh number for the onset of double-diffusive convection. The results of the linear stability analysis, which takes into account both surface tension and double-diffusive effects, are in general agreement with the experimental observation. Results from this stability analysis show that even at  $10^{-4} g_0$ , where  $g_0$  is the gravity level at sea level, the double-diffusive effect is of equal importance as the Marangoni effect.

The effect of gravity modulation on Benard convection was also experimentally studied with compressed air as the working fluid. The test apparatus was mounted on a shaker, which was capable of oscillating up to 2.5 Hz at a fixed amplitude of 10 cm. A series of experiments was conducted, and the results demonstrated convincingly the stabilization effect of gravity modulation on Benard instability. In addition, a transient conduction-convection equation solver has been developed using the Boundary Element Method. This numerical code has been validated and will be applied to study the Marangoni and double-diffusive convection in a fluid layer under microgravity conditions.



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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0

MS Students: 0

PhD Students: 3

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-51

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Journals**

Chen, C.F., Chen, C.C. Effects of surface tension on the stability of a binary fluid layer under reduced gravity. *Phys. Fluids*, A 6, 1482-1490 (1994).

**Presentations**

Chen, C.F. "Effects of surface tension on the stability of a binary fluid layer under reduced gravity." AGU Chapman Conference on Double-Diffusive Convection, Scottsdale, Arizona, November 3-6, 1993.

Chen, C.F. "Solid fraction distribution in a growing mushy layer by computed tomography." 46th Annual Meeting of APS-DFD, Albuquerque, New Mexico, November 21-23, 1993.

Choi, C.Y. and Chen, C.F. "The effect of gravity modulation on convective stability." 10th Arizona Fluid Mechanics Conference, Scottsdale, Arizona, February 25-26, 1994.

Tanny, J. and Chen, C.F. "Experiments on the effect of a free surface on convective instability in a double-diffusive layer." 10th Arizona Fluid Mechanics Conference, Scottsdale, Arizona, February 25-26, 1994.

### *Transport Phenomena in Stratified Flow in the Presence and Absence of Gravity*

PRINCIPAL INVESTIGATOR: Prof. Norman Chigier

Carnegie Mellon University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The strategy of this study is:

1. To separate the effects of molecular and turbulent diffusion from gravitational forces during the mixing of two shear layers.
2. To identify the relevant and characteristic non-dimensional parameters that govern the physical interaction between fluid layers in the range of the proposed experimental conditions.
3. To examine the interaction between buoyant plumes and stratified shear layers, and to study the rates of entrainment into buoyant plumes and stratified shear layers.
4. To study the effects of injecting solid and liquid particles into fluid streams under stable and unstable stratified shear flow conditions.
5. To identify potential instrumentation that could distinguish between molecular and turbulent diffusion in the presence and absence of gravity.

#### TASK DESCRIPTION:

1. Conduct detailed measurements in turbulent mixing layer with velocity and density gradients.
2. Conduct a scoping on the interaction of buoyant heated jets and plumes interacting with stable and unstable stratified shear layers.
3. Develop scaling laws and similarity criteria for comparison with measurements made in the "main" and "reduced" scale experiments.

#### TASK SIGNIFICANCE:

To use the reduced gravity environment to improve the understanding of the fundamental physical processes of mixing between layers of fluids of different densities and velocities. The following are a few matters that are important to this research project:

1. To understand the process of dispersion of pollutants in a highly stable stratosphere and in outer space.
2. To understand the effect of dispersion of pollutants emitted from a space craft at high altitudes.

These results could be used to find the means of controlling or at least reducing pollutions of the atmosphere and the oceans where stratified flows prevail.

#### PROGRESS DURING FY 1994:

Significant progress has been made in 1994. The design and construction of the flow facility for I-g experiments has been completed. A wind tunnel with two separately heated air flows, separated by an insulating splitter plate, has been designed, constructed, and tested. The exit velocity and turbulence profiles have been measured by LDV, and temperature profiles have been measured by thermocouples. Nearly uniform velocity profiles at the exits have

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

been achieved. Heat transfer across the splitter plate results in thermal boundary layers inside the wind tunnel that distort the exit temperature profile. The measured turbulence structure, 127 mm downstream of the nozzle exit, is close to isotropic, and the use of a special turbulence management section, suggested by Dr. Nasser Rashidnia of LeRC, resulted in the successful flow quality downstream in the flow facility.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/93 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-00-71

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Presentations

N.Chigier, M. Xu, D. Squarer, and N. Rashidnia "Transport phenomena in stratified multi-fluid flow in the presence and absence of gravity." Presented at the 2nd Microgravity Fluid Physics Conference. NASA LeRC, Cleveland, OH. June 21-23, 1994. (NASA Conference Publication 3276, pp. 273-278, 1994).

### *Bubble Dynamics, Two-Phase Flow, and Boiling Heat Transfer in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Jacob N. Chung

Washington State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The main objective is to study the effects of external force fields on the nucleation, two-phase bubble dynamics and boiling transport in microgravity. The proposed research seeks to increase our understanding on bubble nucleation and growth on the heater surface, bubble removal from the heater surface by an electric field, an acoustic field or a velocity shear resulting from the relative fluid motion with respect to the surface, and bubble dynamics and heat and mass transport in boiling liquids.

#### TASK DESCRIPTION:

In the experimental portion of the work, visualizations with laser sheet and high-speed photography and heat transfer measurements will be performed in a drop tower at Washington State University and on board a Learjet at NASA Lewis Research Center. The visualization photographs will be analyzed by a digital image analysis system. Also, boiling curves will be developed based on measurements of heat fluxes and surface superheats for various system conditions. Special attention will be focused on the critical heat flux and Leidenfrost point. Specifically, the effects of bulk convective motion will be determined. Quantitative assessment of the effects of impurities and surfactants on the boiling heat transfer rates will also be included. Analytical and numerical modeling will complement the experiments. Perturbation and asymptotic techniques will be applied for low Reynolds number bubble dynamics, linearized bubble stability analysis, and small oscillation of bubbles in microgravity to account for small disturbances and g-jitter.

#### TASK SIGNIFICANCE:

The proposed research seeks to:

1. Increase our understanding of bubble nucleation and growth on the heater surface.
2. Bubble removal from the heater surface by an electric field, an acoustic field or a velocity shear resulting from the relative fluid motion with respect to the surface.
3. Bubble dynamics and heat and mass transport in boiling liquids.

Future spacecraft power systems are likely to have greater power generation and dissipation requirements due to proposed longer space missions and more intensive space activities. Without suffering from weight penalties, two-phase boiling systems are being considered for a wide range of future space applications such as thermal control, propulsion, power generation, and thermal management. The boiling system has the potential advantage of being able to transfer a large amount of energy over a relatively narrow temperature range with a small weight requirements. For example, two-phase systems were once baselined for the space station. However, the thermo-fluid dynamics of two-phase systems in microgravity encompasses a wide range of complex phenomena that were not understood sufficiently for engineering design to proceed.

#### PROGRESS DURING FY 1994:

An old elevator shaft at Washington State University has been transformed into a 2.1-Second Drop Tower. Numerous successful drops have been conducted. This drop tower uses an air bag to decelerate the test package. The drop tower is fully operational.

Both the forced convection microgravity boiling apparatus and the electrostatic microgravity boiling apparatus have been built and tested in the laboratory using the 0.6 second tower. Gold film heaters have been successfully built and tested. To reduce cost the substrate material is polycarbonate instead of quartz. The heaters use a 400 Angstrom gold surface with a power output of  $7\text{W}/\text{cm}^2$ . Results indicate that when the heat flux is relatively low and the subcooling remains relatively high the heat transfer is enhanced in microgravity. At high heat flux levels dryout occurs in microgravity. The results are in good agreement with Merte, et al. Similar tests are planned using the 2.1-Second Drop Tower.

The pool and flow boiling apparatus for the 2.1-Second Drop Tower has been designed and built. Currently calibration and 1-g tests are being conducted, low gravity tests are planned for 1995.

STUDENTS FUNDED UNDER RESEARCH:				TASK INITIATION: 12/92	EXPIRATION: 12/95
BS Students:	0	BS Degrees:	0	PROJECT IDENTIFICATION: 962-24-00-59	
MS Students:	2	MS Degrees:	1	RESPONSIBLE CENTER: LeRC	
PhD Students:	1	PhD Degrees:	0		

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Snyder, T.J., Sitter, J.S., Chung, J.N. "Maiden drops of WSU 2.1 second drop tower with airbag deceleration system". submitted to International Journal of Applied Microgravity Technology, (1994).

Wang, T.C., Snyder, T.J., Chung, J.N. "Transient effects of microgravity on pool and low velocity flow boiling heat transfer". Submitted to Int. J. Heat and Mass Transfer, (1994).

### *Reactive Fluids Experiment: Chemical Vapor Deposition*

PRINCIPAL INVESTIGATOR: Dr. Ivan O. Clark

NASA Langley Research Center (LaRC)

#### CO-INVESTIGATORS:

P.V. Hyer

Lockheed Engineering & Sciences Co.

E. J. Johnson

Lockheed Engineering & Sciences Co.

#### TASK OBJECTIVE:

The research will develop a series of ground-based experimental investigations of the fluid dynamics of chemical vapor deposition (CVD) which will lead to an enhanced understanding of the basic sciences underlying reactive fluid interactions. It will form the basis for a proposal to perform a series of flight experiments necessary to more fully elucidate these scientific principles. This program will use past experience in chemical vapor deposition, non-isothermal flow measurements, numerical modeling of reactive fluid dynamics, and development of instrumentation to carry out the research.

#### TASK DESCRIPTION:

A combined numerical and experimental approach is being used to investigate the CVD process. The experimental approach combines growth of semiconductor materials, the deposition of a model material, and the measurement of the gas flow velocities in the CVD reactor using laser velocimetry. The numerical approach models each of the experimental approaches and uses the experimental results for validation.

#### TASK SIGNIFICANCE:

CVD is an extremely important industrial process. It is widely used not only for the production of semiconductor and insulating materials, but also for optical coatings, wear- and corrosion-resistant coatings, paint pigments, and the production of drawing stock for optical fibers. In addition to the economic importance of these application areas for terrestrial research and manufacturing, they also represent key manufacturing capabilities for future extraterrestrial development. Each of these CVD applications takes place in reactors which have been developed through decades of empirical trial and error. Engineering design capabilities have been limited by the extreme difficulty, under Earth-gravity (1g), of separating the fluid dynamic effects of externally forced convection, buoyant thermal convection, buoyant solutal convection, and internally forced convection due to volume changes arising from both thermal and chemical effects. This research seeks to improve the ability to apply engineering design techniques to this economically important area.

#### PROGRESS DURING FY 1994:

The numerical modeling of InP in three spatial dimensions has been refined with good results. Model/experiment agreement is best when the experimental films are thick. InP film porosity has proven to be a problem on the large-area amorphous substrates. Additional growths are being performed with GaAs and InP substrates to examine substrate effects on measured growth rates.

Laser velocimetry (LV) measurements of the flow velocities in a replica CVD reactor, which duplicates the geometry used for the InP growths, is in progress. The transition from supply manifold to reactor inlet has proven to have a significant effect on reactor flows. Numerical modeling of this reactor geometry has been expanded to include the entry region as well as the effects of thermal gradients on the trajectories of tracer particles used for the LV measurements.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

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TASK INITIATION: 11/90    EXPIRATION: 11/94

PROJECT IDENTIFICATION: 962-24-06-03

RESPONSIBLE CENTER: LaRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Black, L.R., Johnson, E.J., Hyer, P.V., Culotta, P.W., and Clark, I.O. "An improved 3-D numerical model of InP MOCVD with comparison to growth and laser velocimetry experiments." Proceedings of Fluent Inc. 1993 Users' Group Meeting, Burlington, VT, October 5-7, 1993.

*Microgravity Particle Dynamics*

PRINCIPAL INVESTIGATOR: Dr. Ivan O. Clark

NASA Langley Research Center (LaRC)

CO-INVESTIGATORS:

E.J. Johnson  
J.F. Meyers  
S.O. Kjelgaard

Lockheed Engineering & Sciences Co.  
NASA LaRC/RTG/FldMD/NDB  
NASA LaRC/RTG/FldMD/EMB

**TASK OBJECTIVE:**

The objective of this research is to develop the apparatus, numerical models, and practices for enhancing the knowledge of particle transport in laminar fluid flows. Specifically, an enhanced understanding of the roles of thermophoresis (thermal gradient) and Saffman (particle crossing of velocity gradient) effects on particle transport is sought. The proposed research also seeks to identify the critical parameters and instrumentation requirements for subsequent microgravity experiments.

**TASK DESCRIPTION:**

The technical approach is a coordinated numerical and experimental investigation using geometries selected to maximize the scientific return of the research. Gravitational effects in the experimental investigation are addressed through the use of multiple orientations, relative to gravity, of the test chambers. The first step in this investigation is a parametric study to determine the order of magnitude of the competing effects for candidate test chamber geometries. These complex interacting effects include gravity, buoyancy, inertia, viscous drag, particle rotation, electrostatic charges, as well as thermal and velocity gradients. The initial parametric study will ensure a maximum scientific return from the selected geometry. Experimental studies will use the laser velocimetry (LV) and flow visualization systems developed for chemical vapor deposition reactor characterization at Langley Research Center (LaRC). Additional LV and other aerodynamic instrumentation systems are available to this research at LaRC if needed.

**TASK SIGNIFICANCE:**

The proposed research will result in: (1) definition of the requirements and the potential for follow-on flight experiments in transport phenomena, (2) an enhanced understanding of particle transport phenomena in thermal and velocity gradients, and (3) fluid dynamic correction factors for particle-based flow instrumentation for a range of thermophoresis and Saffman environments. The results of this research will be immediately applicable in both unigravity and microgravity for applications such as validation of particle transport theories; correction of wind tunnel research data; and design refinements for chemical vapor deposition reactors, particulate combustors, and clean rooms. In addition, experimental size constraints for microgravity experiments dictate that velocity gradients will exist in laminar flow flight experiments. Hence, Saffman effects will be present to some extent in all microgravity laminar flow experiments with particles.

**PROGRESS DURING FY 1994:**

Steady-state numerical studies of thermophoretic flow patterns have begun. Several shortcomings of the thermophoresis algorithm used in FLUENT have been identified and an improved algorithm has been implemented. Parametric studies of velocity, length, time, thermal gradient magnitude and velocity gradient magnitude likely to be involved in gas-solid multiphase flight experiments have been assessed for 1-100 micrometer particles. The results of this parametric study indicate that flight experiment regimes are both achievable and useful.

A database has been established of particle dynamics research and particle-based instrumentation which will ensure currency of ground and flight experiments associated with this research as well as provide a valuable reference tool for other researchers in this area.



## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/92    EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-06-04

RESPONSIBLE CENTER: LaRC

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BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Proceedings**

Clark, I.O. and Johnson, E.J. "Particle experiments in thermal and velocity gradients." Proceedings of the 2nd Microgravity Fluid Physics Conference, Cleveland, OH, June 21-23, 1994.

### *Studies of Freely Suspended Liquid Crystal Bubbles*

PRINCIPAL INVESTIGATOR: Prof. Noel A. Clark

University of Colorado, Boulder

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The proposed research will develop techniques necessary for the study of levitated liquid crystal bubbles. These techniques will be used in a series of experiments which will seek to study fluid deformation under circumstances of static and dynamic compression. In addition, bubble shape when coupled with a structure, and gas permeability studies will be performed.

#### TASK DESCRIPTION:

These experimental studies require technique developments beyond those which have been used for the study of planar films in 1G. Among the more important ones are (1) a method for inflation, levitation, positional stabilization, of the bubble, (2) transmission video microscopy to monitor bubble shape and polarized reflection video microscopy to probe the local orientation, and (3) methods to change ambient gas pressure while maintaining bubble position.

#### TASK SIGNIFICANCE:

Ultra thin freely suspended liquid crystal films are structures of fundamental interest in condensed matter physics. By eventually conducting this research in microgravity, microscopic forces and stresses on bubbles can be observed in conditions which will accurately simulate two dimensions. Knowledge of thin film structures is necessary to a complete understanding of membrane phenomena, transport processes, fabrication of thin film materials, and other materials related fields.

#### PROGRESS DURING FY 1994:

This new grant was funded on May 16, 1994. The research associate is in the process of setting up the laboratory apparatus necessary for developing and testing the levitation device and procuring the equipment needed for analysis.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 4/94 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-24-05-82

NASA CONTRACT No.: NAG3-1600

RESPONSIBLE CENTER: LeRC

*Fluid Interface Behavior Under Low- and Reduced-Gravity Conditions*

PRINCIPAL INVESTIGATOR: Prof. Paul Concus

University of California, Berkeley

CO-INVESTIGATORS:

Robert Finn

Stanford University

**TASK OBJECTIVE:**

The general objective of this research is to gain a better mathematical understanding of the physical behavior of fluids partly filling a container or otherwise in contact with solid support surfaces, when capillary forces are predominate. Closely interrelated with the mathematical and computational studies are current and planned ground-based and in-space microgravity experiments.

**TASK DESCRIPTION:**

The approach for this project is to pursue parallel theoretical, computational, and experimental studies to explore equilibrium capillary free surface interfaces in a variety of geometrical configurations. The theoretical aspects include both rigorous mathematical studies and numerical computation. In connection with the scheduled USML-2 Glovebox experiment, two configurations of particular interest, for which small changes in container shape or contact angle can give rise to large bulk reorientation of fluid, have been devised and are being studied: 1) a movable wedge and 2) canonical proboscis containers. Experimentation is carried out in collaboration with M. Weislogel at the NASA Lewis Research Center.

**TASK SIGNIFICANCE:**

Knowledge gained from these studies can lead toward a better understanding of surface phenomena of liquids in containers in a low-gravity environment, and in particular to obtaining new information on the significance of contact angle as an intrinsic physical property. Additionally, development of accurate, new methods for measuring contact angle and new insights for managing fluids in space can result.

**PROGRESS DURING FY 1994:**

Work continued on studying a class of "canonical proboscis" containers. These containers are cylinders whose sections have the remarkable property that at a prescribed contact angle a "nearly-discontinuous" transition of bulk fluid occurs, with unbounded rise height over a "proboscis" protrusion; the protrusion can be made relatively as large as desired. The formal proof of this property was completed (jointly with a student, T. Leise), and the detailed behavior of solution surfaces has been investigated (jointly with students A. Chen and F. Zabihi). In addition to a movable wedge container, the proboscis containers will form part of an experiment scheduled for the USML-2 Glovebox. These shapes have possible application to exploiting microgravity for the accurate determination of contact angle: in this regard, double proboscis containers were designed for the experiment, to permit bracketing of the determined contact-angle values. Results of our recent joint IML-2 experiment (D. Langbein, principal investigator) will be useful in the design and interpretation of our USML-2 experiment.

Investigation continued of the problem of the free surface of a liquid partly filling a wedge container (cylindrical container with a protruding corner in its section) in the absence of gravity, when the contact angles on the two sides of the wedge are allowed to have different (constant) values. We have now largely characterized the types of behavior that can occur, some of which are strikingly different from what is possible for the single contact-angle case. In general terms, for the new kinds of behavior the surface normal is necessarily discontinuous at the wedge vertex V. Widely differing behavior is possible within this range, depending on the local boundary curvatures on either side of V and on the particular data. In one explicit example, the existence of a solution with an infinite jump discontinuity at the vertex is demonstrated. Under other conditions bounded solutions can exist, and under

further conditions the solutions must be continuous (this last result follows from recent related work by Lancaster and Siegel). In general, discontinuities must be anticipated, but we can show that whenever a bounded solution exists then every solution is bounded. For particular cases, we have verified the behavior numerically. The precise characterization of all admissible behavior patterns is a principal question that we have started to address.

As part of the design of containers for our earlier USML-1 experiment, the stability of liquid bridges between parallel plates was studied, for the case of equal contact angles on the two plates. This work was extended by a student (L-M Zhou) to the case of unequal angles with some surprising results. In the equal angle case, she gave the first uniqueness criteria, showing that either the bridge is unique or else there are exactly two solutions, of which only one is stable. Zhou's results display a remarkable variety in possible behavior, depending on the prescribed contact angles and volumes.

In related work, the possibility of (tubular) liquid bridges spanning two intersecting planes was investigated by a student, John McCuan. For certain ranges of boundary angles, trivial explicit solutions can be obtained as spheres with two caps cut off. McCuan shows that no other boundary angles can admit any solutions at all. The result is put into relief by an example constructed recently by H.C. Wente, of self-intersecting bridges that achieve angles other than those achievable by spheres. Thus, the physical requirement that the bridge surface not intersect itself is crucial for the result. An open question raised by McCuan's result is whether the spheres are the unique solutions when they exist. His result also suggests that when a spherical bridge exists, if the wedge angle is slowly opened then the bridge should move into the edge, and possibly break into several disjoint drops in accordance with recent results of Langbein. These, and the results in the above paragraphs, suggest possible related experiments in microgravity.

McCuan has also been working experimentally in connection with the "Gravity B" experiment for testing the general relativity theory, which is currently being prepared at Stanford. The vessel is to be partly filled with liquid helium, and it is essential to have precise estimates as to where the helium will be. The problem is too difficult for a reliable theoretical study, but McCuan has used invariance properties of the governing equations to devise and build a ground-based experimental apparatus. The initial experiments have already been completed and have yielded significant information.

Analysis of the data from our USML-1 glovebox experiment continues. This experiment concerned axially symmetric "exotic" containers. The containers, which are in the shape of a right circular cylinder with mathematically derived toroidal-like bulge, have the property that they admit in zero gravity an entire continuum of axially symmetric equilibrium interfaces for the prescribed fluid volume and contact angle. However, it was proved that the entire container can admit no symmetric stable interface; thus the energy minimizing interface (which is known to exist) must necessarily be asymmetric. This behavior was verified in the USML-1 experiment; as the fluid enters the bulge in the container, it appears at first as a spherical cap. But in response to a disturbance it immediately adopts an asymmetric shape. Most of the video tapes of our experiment taken on board USML-1 have now been received, and final analysis of the data awaits arrival of the remaining tapes.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 3/90 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-24-05-66

RESPONSIBLE CENTER: LeRC

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Concus, P. "Equilibrium capillary surfaces: theory and space experiments." American Physical Society, Fluid Dynamics Division Annual Meeting, Albuquerque, Nov. 1993.

Concus, P. "Capillary surfaces in wedge domains: the two angle case." Symp. on Nonlinear Problems, Wichita State Univ., April 1994.

Concus, P. "New results of McCuan and of Zhou." Symp. on Nonlinear Problems, Wichita State Univ., April 1994.

Concus, P. "A-priori bounds for h-graphs." Univ. Oklahoma, Norman, April 1994.

Concus, P. "Capillary Wedges." Symp. on Calculus of Variations, Oberwolfach, May 1994.

Concus, P. "Local and global existence criteria for capillary wedges." Univ. Leipzig, May 1994.

Concus, P. "Gradient estimates for h-graphs." Univ. Cottbus, May 1994.

Concus, P. "The canonical proboscis." Univ. Leipzig, May 1994.

Concus, P. "A-priori gradient and curvature bounds for h-graphs." Univ. Bonn, June 1994.

Concus, P. "Stability criteria for liquid bridges." Univ. Leipzig, June 1994.

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Concus, P. "Equilibrium fluid interface behavior under low- and zero-gravity conditions." Second Microgravity Fluid Physics Conf., Cleveland, June 1994.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Concus, P. "Measurement of capillary contact angle with the canonical proboscis." Drop Tower Days Symp., Univ. Bremen. July 1994.

Concus, P. "Some results from the interface configuration experiment aboard USML-1." COSPAR '94, Hamburg, July 1994.

Concus, P. "Equilibrium capillary surfaces: experiments aboard the space shuttle." Center for Pure and Applied Mathematics Lecture Series, Univ. Calif. Berkeley. Sept. 1994.

*Convection and Morphological Stability During Directional Solidification*

PRINCIPAL INVESTIGATOR: Dr. Sam R. Coriell

National Institute of Standards and Technology (NIST)

CO-INVESTIGATORS:

G.B. McFadden

National Institute of Standards and Technology (NIST)

B.T. Murray

National Institute of Standards and Technology (NIST)

J.R. Manning

National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE:**

During the directional solidification of a binary alloy solute inhomogeneities can arise from both fluid flow and morphological instability. In microgravity buoyancy-driven fluid flow is reduced, and experiments to study the evolution of morphological patterns without the interference of fluid flow may be possible. The goal is to develop an understanding of the interaction of fluid flow with the crystal-melt interface so that solute segregation in solidifying alloys can be controlled in order to obtain materials with superior properties.

**TASK DESCRIPTION:**

Included in this research are:

1. Calculations of cellular morphologies in the absence of fluid flow.
2. Evaluation of the Seebeck voltage for cellular interfaces as a method for monitoring interface morphology in metallic alloys.
3. Linear stability analyses of coupled interfacial and convective instabilities.
4. Calculations of the effects of time-dependent gravitational accelerations (g-jitter) on fluid flow during directional solidification. For growth of a vicinal face at constant velocity, the effect of anisotropic interface kinetics on morphological stability has been calculated for a binary alloy. Anisotropic kinetics give rise to traveling waves along the crystal-melt interface, and can lead to a significant enhancement of morphological stability.

This ground based research will focus on providing theoretical interpretation and guidance for a series of space experiments to be carried out by J.J. Favier, R. Abbaschian, and colleagues on tin-bismuth alloys using the MEPHISTO apparatus and by K. Leonartz and colleagues on succinonitrile-acetone alloys.

**TASK SIGNIFICANCE:**

This comprehensive research on the directional solidification of binary alloys will determine the processing conditions required to eliminate microsegregation of impurities and produce alloys with desirable microstructures and improved properties. This research will potentially contribute to the important and challenging goal to develop and transfer to all of the consortium members (of NIST, US industry, and NASA) technology that will provide for the rapid design and prototyping of new precision cast parts, enhance product quality, and reduce rejection rates.

**PROGRESS DURING FY 1994:**

The solute mass transport that may lead to morphological instabilities in directional solidification has been described within the framework of the nonlinear thermodynamics of irreversible processes. Specific calculations have been carried out for tin-bismuth alloys for two models of the concentration dependence of the thermodiffusion flux. Depending on the sign of the thermodiffusion coefficient, morphological stability may be increased or decreased.

We have investigated the effect of anisotropic kinetics on the morphological stability of a pure material growing at constant velocity into a super cooled melt. The kinetic anisotropy is based on the motion of elementary steps and the stability of a planar interface with respect to the formation of macro steps or step bunching is considered. Kinetic anisotropy and capillarity stabilize the interface, while the temperature gradient in the melt is destabilizing. Linear stability calculations for dislocation and nucleation controlled growth of salol indicate that anisotropic kinetics provides stabilization for sufficiently low growth velocities for crystals of practical dimensions. Although the critical crystal size depends on the growth velocity, for materials like salol, instability may occur for dimensions larger than tens of centimeters, while for materials like germanium the critical size is less than a centimeter.

The effect of a parallel shear flow and anisotropic interface kinetics on the onset of instability during the directional solidification of a binary alloy at constant velocity has been calculated. A shear flow (linear Couette or asymptotic suction) parallel to the crystal-melt in the same direction as the step motion decreases interface stability in that the critical solute concentration decreases. A shear flow counter to the step motion enhances stability for small shear rates; for larger shear rates, an additional mode of instability occurs and the critical solute concentration slowly decreases with shear rate.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-40

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

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*Microphysics of Close Approach and Film Drainage and Rupture During Drop Coalescence*

PRINCIPAL INVESTIGATOR: Prof. Robert H. Davis

University of Colorado, Boulder

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The overall objective of this research is to develop a comprehensive theoretical model of the relative motion, film drainage, and film rupture leading to coalescence of interacting drops dispersed in an immiscible fluid. Relative motion due to gravity, thermocapillary migration, and attractive van der Waals forces is considered. This research is concerned with the microphysics of coalescence and focuses on the near-contact interaction of a drop approaching a second drop or a surface or interface

**TASK DESCRIPTION:**

The overall goal of this microphysical research is to predict deformation, film drainage, and collision rates using fundamental theoretical analyses. The novel method used couples lubrication theory in the narrow separation gap and boundary integral theory for the drop phase. Matched asymptotic expansions are used for small times (small drop deformations) and long-times (draining film regions).

The research program is divided into three components directed at meeting the goal:

1. Near-contact relative motion for nearly spherical drops: The rate of approach and the onset of deformation and film drainage are examined for gravity-driven and thermocapillary-driven motion.
2. Evolution of drop deformation during film drainage: As the drops move closer, the natural evolution of the shape of the thin film separating them is predicted, as is the rate at which this film drains.
3. Film rupture due to van der Waals forces: When the rate-limiting coalescence step of film drainage causes an unstable film to become very thin, then attractive van der Waals forces pull the drop interfaces together and cause rupture. The rupture time and the rupture mode are determined as functions of the system parameters.

**TASK SIGNIFICANCE:**

Drop interactions and coalescence play key roles in a variety of natural and industrial phenomena, including liquid-liquid extraction, raindrop growth, multiphase flow, and processing of bimetallic melts within the liquid-phase miscibility gap. In liquid-liquid extraction of separation, it is advantageous to promote interactions and coalescence for efficient and faster operations, while in the processing of bimetallic materials coalescence must be controlled so that the two materials are uniformly dispersed in the final product. This research aims to provide a better understanding of the fundamental factors governing drop interactions and coalescence.

**PROGRESS DURING FY 1994:**

The collision rate of small drops in linear flow fields was solved for large Peclet numbers and small Reynolds numbers. Simple shear flow and uniaxial extensional or compressional flow were considered. The analysis makes use of the lubrication theory for two drops in near-contact relative motion developed previously. Various trajectory types were identified (open, closed, semi-closed, collision), and it was shown that collisions do not occur in simple shear flow for size ratios smaller than a critical value.

The related problem in which a spherical drop or bubble captures smaller drops or particles by diffusive, convective, or convective-diffusive mechanisms was solved under creeping flow conditions. This problem has important environmental applications in the microflotation of colloids and oil droplets from aqueous streams, and biotechnology applications in aqueous biphasic extraction of cells and cellular components.

The thermocapillary motion of drops nearly in contact has been studied when two drops are non-conducting or when a non-conducting drop approaches a contact temperature surface or an interface. These results have been incorporated in trajectory analyses to predict thermocapillary induced collision rates. When the drops are highly conducting, the relative velocity reverses sign and coalescence is impossible.

For combined motion due to thermocapillary and gravitational effects, it has been shown that collisions are reduced or eliminated upon antiparallel alignment of the thermocapillary and gravitational motions in a certain range of their relative magnitude. For combined Brownian and gravitational or shearing motion, a numerical analysis for arbitrary Peclet numbers shows that synergistic effects greatly increase the pairwise collision rate of drops.

STUDENTS FUNDED UNDER RESEARCH:				TASK INITIATION: 5/91	EXPIRATION: 5/94
BS Students:	0	BS Degrees:	0	PROJECT IDENTIFICATION: 962-24-00-35	
MS Students:	0	MS Degrees:	0	NASA CONTRACT NO. NAG 3-1277	
PhD Students:	2	PhD Degrees:	1	RESPONSIBLE CENTER: LeRC	

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*Phase Segregation Due to Simultaneous Migration and Coalescence*

PRINCIPAL INVESTIGATOR: Prof. Robert H. Davis

University of Colorado, Boulder

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this research is to understand the interaction and coalescence of bubbles and drops due to thermocapillary and gravitational effects. Modeling is performed via population dynamics balances to predict the rate of phase segregation under the collective or individual action of the driving forces.

**TASK DESCRIPTION:**

Significant effort is being devoted to the development and performance of ground-based experiments. The trajectories of interacting drops and the rate of phase segregation are being measured in a transparent immiscible liquid system under isothermal conditions.

Theoretical work is also being performed to predict the macroscopic phase separation and drop-size distributions due to buoyancy and thermocapillary motion and coalescence of immiscible dispersion of drops by solving the general population dynamics equations retaining both spatial and time dependencies.

**TASK SIGNIFICANCE:**

This research on phase segregation attempts to provide a predictive tool to determine the size distributions of drops in a heterogeneous immiscible fluid mixture. Inputs from other fundamental studies on drop interactions and coalescence are used in population dynamics models to predict the size distributions. This research will ultimately be of use in materials processing, food and beverage processing, and perhaps in the biotechnology industry.

**PROGRESS DURING FY 1994:**

The population dynamics equations for homogeneous dispersions have been solved for droplet growth due to the separate effects of Brownian, gravitational, and thermocapillary motion and coalescence, and due to the combined effects of thermocapillary and gravitational motion and coalescence. For thermocapillary motion alone, highly conducting drops experience thermocapillary repulsion, in which the smaller drop moves away from a nearby larger drop due to distortions in the temperature field, thereby preventing coalescence. For combined, antiparallel gravitational and thermocapillary motion, a collision-forbidden region of parameter space is possible due to the faster decay with separation distance of the thermocapillary interaction of two drops of bubbles than of the gravitational interaction.

A computer code has been developed for nonhomogeneous dispersions undergoing simultaneous phase separation and motion and coalescence due to gravity. The rate of phase separation initially increases due to coalescence and then decreases due to the larger drops moving out of the suspension. A key dimensionless parameter, representing the ratio of sedimentation and coalescence time scales, governs the process.

Experiments to observe drop coalescence and phase segregation due to gravity have been performed with 1,2-propanediol drops in dibutyl sebacate and with an aqueous biphasic mixture of 1% dextran (MW = 500,000) and 5.5percent polyethylene glycol (MW = 8,000) by weight. Results for the phase separation rate versus time are in good agreement with the theory and may be used to infer values for the composite Hamaker constant which represents the strength of the attractive van der Waals forces.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 12/92    EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-05-39

NASA CONTRACT No.: NAG3-1389

RESPONSIBLE CENTER: LeRC

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#### Presentations

Davis, R.H., Wang, H., and Hawker, D. "Phase segregation due to simultaneous migration and coalescence." NASA Conf. Pub. No. 3276: 2nd Microgravity Fluids Conference. 101-106. 1994.

#### Thesis

Wang, H. "Modeling and experimental studies of drop coalescence and phase separation." PH.D. Thesis. Univ. Colorado. 1994.

*Interaction and Aggregation of Colloidal Biological Particles and Droplets in Electrically-Driven Flows*

PRINCIPAL INVESTIGATOR: Prof. Robert H. Davis

University of Colorado, Boulder

CO-INVESTIGATORS:

P. Todd

University of Colorado, Boulder

M. Loewenberg

University of Colorado, Boulder

**TASK OBJECTIVE:**

The objective of this research is to develop a fundamental understanding of aggregation and coalescence processes during electrically-driven migration of particles (or cells) and droplets.

**TASK DESCRIPTION:**

The research includes the following tasks:

- Development of a theoretical description of electrically-driven particle aggregation by computing the relative velocity between two particles in near contact with hydrodynamic, electrokinetic, van der Waals, and electrostatic double-layer interactions, and by predicting the stability conditions and the rate of pair-wise aggregation in a semi-dilute suspension;
- Formulation of a description of electrically-driven drop coalescence by developing a simplified electrokinetic description of a charged fluid interface under thin double-layer conditions, and performance of initial computations for the interactions between a pair of electrically-driven droplets; and
- Observations of electrically-driven aggregation and coalescence by conducting terrestrial experiments to test the theoretical description of electrically-driven particle aggregation and observe electrically-driven droplet coalescence, with the results leading to future design of a flight-based experiment to gather quantitative observations for testing a theoretical description of electrically-driven droplet coalescence.

**TASK SIGNIFICANCE:**

The fundamental study of particle aggregation in electric fields is expected to have practical application to electrically-controlled cell flocculation for cell separation and recycle in space-based bioreactors, where gravity cannot be employed as previously done. Similarly, research conducted on drop interactions and coalescence is expected to provide an understanding of electrically-driven demixing of two liquid phases, such as those encountered in biphasic aqueous extraction of biological cells and molecules under reduced gravity when buoyancy-driven demixing is weak. Finally, the theoretical descriptions of two charged, migrating particles or drops are expected to have general scientific and engineering value.

**PROGRESS DURING FY 1994:**

A theoretical description of the near-contact interaction of two particles in an electrical field has been completed. Near-contact particle motion is important because it has a large effect on pairwise collision rates and provides the essential physics of the aggregation process.

The new results for near-contact interaction of particles during electrophoresis were combined with previous results for moderate and large separations in a trajectory analysis to predict pairwise aggregation rates of charged particles with differing zeta-potentials. In addition to hydrodynamic interactions, attractive van der Waals forces and repulsive electrostatic forces were included in the analysis. The near-contact interactions were also used to generate the stability diagram for heterogeneous suspensions in an electric field. The key result is that particle aggregation under an electric field occurs more easily and efficiently than under a gravitational field.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Experiments were performed to measure the electrophoretic mobilities and zeta-potentials of human and rabbit blood cells for a wide range of ionic strengths. The results provide the ranges of salt concentrations for which mobilities differ so that relative motion and aggregation are possible. Microvideo experiments to observe aggregation have been initiated, but quantification is difficult since even moderate concentrations (1% cells) are optically opaque, whereas very dilute suspensions have very low aggregation rates.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 3/93 EXPIRATION: 3/96  
PROJECT IDENTIFICATION: 962-24-08-10  
NASA CONTRACT NO.: NAG8-945  
RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Proceedings

Davis, R.H., Nichols, S.C., Loewenberg, and Todd, P. "Electrophoretic interactions and aggregation of colloidal biological particles." Proceedings of the 2nd Microgravity Fluid Physics Conference. NASA Conference Publication 3276, 379-384 (1994).

*Theory of Solidification*

PRINCIPAL INVESTIGATOR: Prof. Stephen H. Davis

Northwestern University

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

## TASK OBJECTIVE:

This work concerns our effort to understand - on a quantitative level - how various factors affect the morphology of solidification fronts and, hence, the resulting microstructures of the solidified material.

## TASK DESCRIPTION:

In the approach, nonlinear stability theory, asymptotic, and numerical methods are used to investigate the stability of the coupled systems describing the directional solidification of binary systems from the melt.

## TASK SIGNIFICANCE:

The project aims at the theoretical prediction of microstructure in crystalline materials that would allow the a priori "design" of new materials. We will endeavor to answer the following central scientific question: How and under what conditions can crystals be grown in microgravity ( $\mu g$ ) with different and "better" properties than those grown on Earth?

## PROGRESS DURING FY 1994:

Progress has been made in many areas of solidification including linear and nonlinear stability theories of rapid solidification in which thermodynamic nonequilibrium effects at the interface are allowed. The dynamics of trijunctions during phase transition, the emergence of islands in epitaxial growth and the shear stabilization of morphological fronts have been analyzed, and promising results obtained.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 10/87 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-25-05-16

NASA CONTRACT NO.: NAG3-747

RESPONSIBLE CENTER: LeRC

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*Microgravity Foam Structure and Rheology*

PRINCIPAL INVESTIGATOR: Prof. Douglas J. Durian

University of California, Los Angeles

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this research is to investigate the origin of the most striking and least understood rheological properties of foam by measuring elastic and flow behavior for a sequence of aqueous foams with increasing liquid content. The microscopic structure and dynamics of the foams will simultaneously be characterized by recently developed multiple light scattering techniques.

**TASK DESCRIPTION:**

Foam structure and dynamics will be measured directly and non-invasively through development and use of novel multiple light scattering techniques such as Diffusing Wave Spectroscopy (DWS). Foam rheology will be measured in a custom rheometer which allows simultaneous optical access for multiple light scattering. Microgravity conditions will ultimately be required to eliminate the increasingly rapid gravitational drainage of liquid from in between gas bubbles as the liquid:gas volume fraction is increased toward the rigidity-loss transition.

**TASK SIGNIFICANCE:**

This experiment will constitute the first measurement of how the surprising solid-like elastic quality of foam vanishes as the volume fraction of liquid is increased. The simultaneous measurements will also permit the first quantitative correlation of macroscopic rheological behavior with the underlying microscopic structure and dynamics, thus providing new insight into the origin of the dual solid/liquid nature of foams.

**PROGRESS DURING FY 1994:**

In the current funding period we have made important progress both in terms of the development of multiple light scattering techniques and their application to a model foam system. As for the former, analysis of multiple light scattering data crucially assumes that photon propagation can be described by a diffusion approximation with source and boundary terms set by a phenomenological penetration depth and extrapolation length, respectively. While the accuracy of this approach can in principle be no greater than about 1%, far greater errors are introduced in practice due to inappropriate treatment of source and boundary terms. I have determined how to average over the penetration depth, and how to experimentally deduce the extrapolation length. My objective, which is now nearly achieved, is to put Static Transmission (ST) and Diffusing-Wave Spectroscopy (DWS) theories on firmer theoretical ground and advance the reproducibility and accuracy of their application down from the 10% level to the 1% level. This is crucial if ST and DWS are to be truly quantitative probes of foam structure and dynamics. In addition, I have introduced a new optical configuration for DWS experiments which provides for significantly better photon-counting statistics and is less susceptible to systematic errors from imperfect laser beam conditioning.

To move towards our goal of simultaneous light scattering and rheology measurements, graduate student Anthony Gopal and I have recently completed a series of experiments on the response of foam dynamics to shear deformations in a sample cell consisting of two parallel glass plates, one of which can be translated at uniform velocity in order to shear the foam. The normalized electric field autocorrelation function is found to decay nearly exponentially with time. The value of the cumulant is set by the ratio of sample cell thickness to the transport mean free path of light and the rate of rearrangement events. The rate of rearrangement varies with the applied shear strain rate. If the applied shear strain is small compared to the rate of rearrangements in the quiescent foam, then the measured rate is unchanged. If it is larger, however, the rearrangement event rate increases and appears to be equal to the shear strain rate. At still higher strain rates, the shape of correlation function begins to change and it appears that the foam is beginning to melt; in this regime, the duration of rearrangement events may be the significant time scale.



Following the cessation of an imposed shear strain, we have also measured intensity fluctuations and find that the rate of rearrangements decreases if the total shear strain suffered is greater than about 5%. For strains greater than on the order of one, we find that the rate of rearrangement decreases by a factor of roughly two and remains depressed until the foam coarsens significantly. This implies that there are important correlations between neighboring bubbles which result from the coarsening process and that this self-organized structure is naturally marginally stable.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-73

NASA CONTRACT NO.: NAG3-1419

RESPONSIBLE CENTER: LeRC

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*The Influence of Gravity on Nucleation, Growth, Stability and Structure in Ordering Soft-Spheres*

PRINCIPAL INVESTIGATOR: Prof. Alice P. Gast

Stanford University

CO-INVESTIGATORS:

Dr. David J. Pine

Exxon Research & Engineering

**TASK OBJECTIVE:**

While previous investigations of colloidal crystals have focused primarily on the structures produced in the ordered arrays, we intend to probe the dynamics of the crystallization process, in particular, focusing on the influence of the gravitational field on ordering. In this study, we will use a novel new light scattering experiment, known as diffusing wave spectroscopy, allowing us to monitor particle motion and ordering in an otherwise turbid suspension. This approach will provide the means to study, on a particle level, a model ordering process in situ. The application of diffusing wave spectroscopy can thus be viewed as an important new noninvasive experimental probe of interparticle dynamics.

**TASK DESCRIPTION:**

Among the attractive features of aqueous colloidal suspensions are long-range Coulombic repulsions diminishing the influence of hydrodynamic interactions between particles. In this system we can observe and provoke homogeneous nucleation, bcc or fcc lattice formation, defects, twinning and a crystallization growth instability analogous to those observed in molecular systems. These phenomena motivate and provide a means for us to investigate the role of interfacial tension, transport and gravity in this process. A key feature in this study is the use of density matching conditions to study the late stages of crystallite growth and ripening without the complicating factors arising from their sedimentation. In order to investigate the influence of a gravitational field on ordering in soft-sphere colloidal suspensions we propose to begin with ground-based studies of polystyrene particles of size ranging from 100 to 500 nm suspended in mixtures of hydrogenated and deuterated water. Choosing an appropriate mixture of D<sub>2</sub>O and H<sub>2</sub>O we can render the suspension neutrally buoyant. In this fashion we will be able to monitor nucleation rate, growth, and ripening under a variety of gravitational conditions. Future studies on more dense colloidal particles of technological importance would require actual low gravity experiments.

**TASK SIGNIFICANCE:**

An additional feature of this research will be an improved understanding of the transport of multiply scattered light through complex, multiphase systems. Our combination of experiments and theoretical treatments is of fundamental importance in the understanding of phase transitions and for further applications of diffusing wave spectroscopy. The interest in colloidal systems as important materials themselves and as model molecular systems is only beginning to expand to phase transition dynamics. This study will provide a definitive test of the nature of ordering dynamics, and its sensitivity to gravity. The link to molecular processes will have important implications for future studies of gas-liquid and solid-fluid transition kinetics.

**PROGRESS DURING FY 1994:**

In a previous phase of our work we used diffusing wave spectroscopy (DWS) to study the dynamics in strongly interacting suspensions of colloidal particles. A detailed description recently appeared in the J. Chem. Phys. (101 (6), 15 Sept. 1994, p. 4975-85). In that work we qualitatively related the interparticle structure to the particle dynamics by measuring the fluctuations in multiply scattered laser light (DWS). The current phase of our work focuses on understanding the dynamics in ordered arrays of colloidal particles through similar light scattering techniques.

Typically colloidal crystals are composed of many small (< 1mm) randomly oriented crystallites with disordered grain boundaries. This makes unambiguous interpretation of the photon scattering rather difficult. To circumvent this difficulty, we have built a Couette cell in which the shear flow can be used to order the suspensions into

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

aligned macro-crystalline domains which we then use in our experiments. We are currently performing experiments on crystals at different aqueous ionic strengths.

This proposal was recently awarded under the 1993 NRA. Funding for this research began July, 1994.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1.3

TASK INITIATION: 7/94 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-24-00-83

RESPONSIBLE CENTER: LeRC

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*Fluid Mechanics of Capillary Elastic Instabilities in the Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. James B. Grotberg

Northwestern University

CO-INVESTIGATORS:

Prof. David Halpern

University of Alabama

**TASK OBJECTIVE:**

The primary objective of this work is to study the fluid mechanics of pulmonary airway closure in the microgravity environment. This task is also to study the stability of liquid lined flexible tubes with surfactants in the liquid.

**TASK DESCRIPTION:**

The task proposed to study coupled fluid-elastic instabilities of a flexible tube coated internally with a thin liquid film by both theoretical and experimental means. The principal investigator will analyze a mathematical model of post-closure filling flows and re-opening phenomena using a similar approach as the closure problem. An experiment of a flexible tube containing a thin liquid layer on its wall and a liquid core of a much smaller viscosity but similar density is also proposed.

**TASK SIGNIFICANCE:**

This research has three important applications: 1) closure and reopening of small airways in the lung, 2) annular extrusion processes, and 3) two-phase flow in porous and poro-elastic materials.

**PROGRESS DURING FY 1994:**

This grant formally began on June 29, 1994. Research has been conducted to address the capillary-elastic instabilities of liquid-lined flexible tubes. An experimental apparatus is used to create a mechanical model of flow in airway tubes. Pulmonary airways are flexible tubes which are lined with a thin liquid film. A small glass tube of ID=0.58 mm is viewed under a microscope-camera configuration. The annular film is created by manually filling the tube with oil from a syringe. Results from experiments using oil films and a water core in a rigid tube have been performed, which appear to match well with theoretical values derived by Halpern & Groterg for higher Capillary number (CA) values; continued experiments will be performed to match with results over full range of capillary numbers. Research using flexible-walled tubes is planned to follow. The research experiments are being conducted by a graduate student, Ms. Karen Cassidy, who is partially supported by this grant.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-00-84

RESPONSIBLE CENTER: LeRC

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*Effects of Convection on the Thermocapillary Motion of Deformable Drops*

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PRINCIPAL INVESTIGATOR: Prof. Hossein Haj-Hariri

University of Virginia

CO-INVESTIGATORS:

Prof. A. Borhan

Pennsylvania State University

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**TASK OBJECTIVE:**

The objective of this theoretical effort is to extend the understanding of thermocapillary motion of drops by including drop deformations. The convective transport of momentum and energy as well as the effects of container walls will be accounted for by the work.

**TASK DESCRIPTION:**

The focus of the initial effort is on the following tasks (a) theoretical work on the boundary-integral formulation for the axi-symmetric motion and deformation of a drop in a cylindrical container, (b) finite-volume formulation of the same problem using domain-mapping techniques, and (c) analysis of the transport of a color function to demarcate the drop boundary.

**TASK SIGNIFICANCE:**

This research will provide information on the motion and shapes of drops driven by interfacial tension gradients. A novel feature of this research is that to facilitate the modeling of this phenomenon using a computer, the sharp boundary between the drop and the surrounding liquid is "smeared" in the computations. The anticipated outcome of this research is the speed and shapes of the drops under various conditions and an enhanced understanding of how much the interface can be smeared before the results are intolerably degraded.

**PROGRESS DURING FY 1994:**

In one year we developed a 2D/axisymmetric version of this Navier-Stokes solver which demarcates the fluid phases using a passively convected color having a constant value over each fluid phase and varying abruptly at their interfaces. The fidelity of the code was enhanced by implementing a quad-tree refinement of the grid, as well as, a second-order TVD scheme for the solution of the color equation. During the past year this code was used to study several physical problems and at the same time work started on the development of the three-dimensional version of the code. Progress in each area is reported below:

## 1. The following computational tools have been developed:

a) *Color function (3D)* - - The extension from 2D to 3D was a major undertaking in the sense that we completely changed the code and ported our platform from Fortran 77 to Fortran 90. This step was essential for the implementation of the oct-tree refinement of the grid in the vicinity of the interface. The dynamic array dimensioning and the link-list structure helped immensely in the development of the data structure for this adapted, unstructured grid. The TVD scheme was also extended to 3D and is now tested. The fluid dynamical equations are being coded presently and should be ready and tested in three to four weeks. The quality of this code is better than any other due to the painstaking attention to the importance of high-resolution near the interface. No other technique that we are aware of can claim to produce resolutions compared to our technique. The high accuracy translates into results of quantitative significance as well as qualitative significance. One drawback is the unsuitability of the nonstructured grid for vectorization; a problem under current consideration.

b) *Domain mapping* - - In year one a second Navier-Stokes solution method using the mapping of the drop or the interface of interest onto a known and simple geometry of similar topology was developed. This year it has been extended to be based on a spectral technique so that more accurate studies addressing stability topics such as weakly nonlinear hydrothermal instabilities can be addressed.

2. The following analytical study was conducted:

a) *Effect of smearing* - - Given that techniques such as ours, or those based on interface tracking, have suddenly become extremely popular, we performed a study to assess the effect of smearing of properties on various gross quantities such as the migration velocity, etc. It was shown that whereas the results are rather insensitive to the smearing of the fluid transport properties (such as viscosity and density), they are quite sensitive to the smearing of the interfacial force into an equivalent body force.

3. The following problems are being studied:

a) *Thermocapillary motion of a drop in unbounded liquid* - - Using the 2D color-function approach the quantitative effects of non-zero Re and Ma were observed. The inertial effects are substantial even in the cases where the drop undergoes very minor deformations.

b) *Interfacial-driven motion in an open cavity* - - Using domain mapping the time-accurate flow in an open three-dimensional cavity subjected to interfacial temperature gradients is simulated.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-00-45

RESPONSIBLE CENTER: LeRC

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Shi, Q., Haj-Hariri, H. and Borhan, A. "Thermocapillary motion of deformable drops." 46th Annual Meeting of the Division of Fluid Dynamics of the APS, Albuquerque, NM, November 1993.

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*Interfacial Transport and Micellar Solubilization Processes*

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PRINCIPAL INVESTIGATOR: Prof. T. A. Hatton

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this research is to develop and implement a new diffusion cell for the fundamental investigation of the effects of surfactants on the interfacial transport of solutes between two phases. In the one case, the concern will be the retardation effect the surfactant monolayer has on solute transfer at conditions below the critical micelle concentrations. This work will assist in determining the effects of interfacial compressibility and solute and surfactant structure on the activation barriers to transport. In the other case, the rates of formation of micelles and reversed micelles at the interface will be investigated. These, too, will be affected by the prevailing solution conditions. To date, this type of study has been hindered because the techniques used for measurement of interfacial transport processes are prone to artifacts associated with ill-defined hydrodynamic conditions.

**TASK DESCRIPTION:**

During this task period, we have concentrated on developing a new diffusion cell approach for the measurement of interfacial transport and solubilization rates without introducing hydrodynamic effects that can complicate data interpretation and analysis. The technique relies on the fact that many fluorophores can be bleached irreversibly when exposed to high intensity light sources for brief periods of time. The rate at which fluorescence is recovered in the bleached zone depends on the diffusion of fluorophores from outside this region. Our task has been to set up the equipment, to integrate the various components, and to establish a methodology for data analysis. A testing program to identify the important parameters ranges over which a space-based experimental study would be beneficial is to be undertaken.

**TASK SIGNIFICANCE:**

Interfacial transport and solubilization processes are important in many industrial, commercial and home operations, ranging from large-scale reaction and separation processes to detergency in dishwashing and laundry applications. New separation and reaction technologies based on surfactant self-assembly show promise for reducing the potential for environmental contamination in many industrial processes, or for remediation of already contaminated resources. Through the development of techniques to measure the rates at which the processes of interest occur, and relating these data to the chemical properties of the systems under study, we will be in a position to design more effectively the surfactants for specific new applications. In some cases, gravity-driven convective currents will complicate the data analysis and interpretation, and this establishes the need to conduct experiments under the low-gravity conditions of a space-based experimental program.

**PROGRESS DURING FY 1994:**

The new system using fringe recovery after photobleaching (FRAP) has now been set up, and all components integrated to enable the efficient gathering of images of the new concentration profiles on either side of the interface as a function of time. Video enhancement of these images, and subsequent quantification of the concentration profiles as a function of time, are now possible, paving the way for a detailed study of the interfacial transport and solubilization rates for various water-oil-surfactant systems. The conditions of fluorophore concentration/bleaching intensity which give rise to unstable density gradients are beginning to be delineated.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 2

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 962-24-08-11

NASA CONTRACT No.: NAG8-951

RESPONSIBLE CENTER: MSFC

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*Critical Phenomena, Electrodynamics, and Geophysical Flows*

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PRINCIPAL INVESTIGATOR: Dr. John Hegseth

University of New Orleans

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

To demonstrate the feasibility of using fluid near its critical point as a compressible fluid for an electroconvection experiment.

**TASK DESCRIPTION:**

Create an experimental system where a spherically symmetric density gradient is established in a compressible fluid. Such a system called Compressible Geophysical Flow Experiment (CGFE) will mimic the property of planetary fluid flows. In order to understand how such an experimental system works, knowledge of elements from fields such as critical phenomena, electrodynamics and compressible fluid dynamics have to be brought together.

**TASK SIGNIFICANCE:**

This experimental work will help the understanding of the Geophysical flows and their characteristics and direct consequences.

**PROGRESS DURING FY 1994:**

This new task was initiated on 8/22/94. We have begun the process of setting up the laboratory, designing the apparatus, and the temperature and pressure control systems, and testing the feasibility of the mechanical driving. In addition, we are testing a high voltage A.C. power supply and ordering equipment and parts. The benefits of studying previous systems will aid in the important design decisions needed for manufacturing the cell. To date we have purchased a frame grabber, optical diagnostic equipment, parts for the cell, and the temperature control system.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 8/94 EXPIRATION: 8/96

PROJECT IDENTIFICATION: 962-24-00-85

RESPONSIBLE CENTER: LeRC

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*Thermocapillary Instabilities and g-jitter Convection*

PRINCIPAL INVESTIGATOR: Prof. George M. Homsy

Stanford University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this study are:

1. To study the stability characteristics and to establish the conditions under which thermocapillary flows become three-dimensional, time-dependent, and/or chaotic.
2. To analyze the g-jitter effects on surface-tension driven flow.

**TASK DESCRIPTION:**

Complimentary experimental and computational studies will be conducted to characterize the transport phenomena induced by surface tension gradients or transient accelerations. Experimentally, measurements of various flow variables will be carried out to elucidate the mechanism of the instability and the dependence of instability on various parameters of the problem. The range of Marangoni numbers will be extended to study the transitions to time-dependent flows, and perhaps to turbulence. Linear stability analyses, using a combination of inverse iteration and Lanczos methods, will be conducted to help elucidate the connection between driven cavity problems and thermocapillary convection problems.

**TASK SIGNIFICANCE:**

The results from this study should enhance the understanding of the basic forces acting on fluids in a space-flight environment, and the accompanying fluid response. Such an understanding can contribute directly to better the fluid handling/management technologies. This can help improve planning and execution of any space experiment that has fluids involved, thus maximizing the science/technology return pursued by these space-flight experiments, which encompasses a wide range from materials processings to biomedical research.

**PROGRESS DURING FY 1994:**

This project involves study of two distinct problems in microgravity convection: thermocapillary convection and its stability, and g-jitter convection.

In FY94, progress on the former has consisted of completion of computational studies of the stability of the lid driven cavity, the results of which have been published. A manuscript on our experiments on the stability of combined buoyancy-thermocapillary convection in a cavity has been submitted for publication in the Physics of Fluids and has been favorably reviewed. An outstanding postdoctoral student from Prof. Tom Mullins' group at Oxford will join us in early October. He will continue our experimentation on thermocapillary cavity flows.

Studies in g-jitter convection have been conducted by a PhD student, Ahmed Farooq, who has just successfully completed his PhD thesis defense. A first paper on g-jitter convection in a cavity has appeared and his thesis is complete. A second paper dealing with instabilities and chaotic convection accompanying high amplitude g-jitter convection is in the final stages of preparation.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 962-24-05-48

RESPONSIBLE CENTER: LeRC

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*Kinetic and Transport Phenomena in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. David Jasnow

University of Pittsburgh

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Theoretical research will be carried out in several areas which involve kinetic and transport phenomena in a microgravity environment. Specifically, attention will be paid to a number of two-phase phenomena in which thermocapillarity effects play an important role. These include, kinetics of phase separation, coupled diffusing fields (e.g., temperature and concentration) and the motion of and transport through two-phase interfaces.

**TASK DESCRIPTION:**

Methods proposed include modeling of conserved order parameter dynamics in two-phase situations at the coarse grained or semimicroscopic level. Optimized codes are being developed to carry out the modeling. Coupling of order parameter to hydrodynamic fields and temperature fields are included. Thermocapillary effects such as thermomigration of drops and coalescence are observed, characterized and the underlying physics analyzed. Modeling will also be performed using techniques developed in the study of dynamical systems.

**TASK SIGNIFICANCE:**

The modeling at the coarse grained, semimicroscopic level allows description of phenomena over length scales ranging from the correlation length on up to semi macroscopic sizes. Local thermodynamics is included using the coarse grained free energy. The interface motion is naturally tracked and surface tension dependence on temperature and other parameters is naturally included in the specification of the coarse grained free energy. Hence universal features of transport in complex situations can be extracted, and, for example, examination of phenomenological boundary conditions can be performed at a somewhat more microscopic level.

Modeling from the perspective of dynamical maps abandons the familiar partial differential equations of the subject, but preserves the essential physics, symmetries, etc. Here, if successful, a substantial improvement in computational efficiency can be achieved, at the cost of being able to compute specific material-dependent quantities.

**PROGRESS DURING FY 1994:**

The main effort under this theoretical project during this period has been to observe thermocapillarity effects such as thermomigration in two-phase systems, within a coarse-grained, semimicroscopic description, to characterize the phenomena and to understand the physical mechanisms for them. There are three main areas of progress: (a) The response and dynamics of a two-phase interface, in a coarse grained description, to a temperature gradient; (b) Thermomigration phenomena and phase separation kinetics in a temperature gradient, where the dominant effects are diffusion driven; and (c) A study of two-dimensional capillarity driven motion of single and multiple droplets, and phase separation kinetics, including hydrodynamical effects.

Project (a) was designed to gain insight to the effect of a temperature gradient on a two-phase interface. Both using analytic approaches and numerical relaxation methods for the dynamics of a nonconserved order parameter, we have determined the motion of an interface for a nonconserved order parameter under a temperature gradient, and have analyzed the free energy landscape for an interface under different boundary conditions.

In (b) we observe, and analyze using arguments derivable from the coarse-grained free energy for the system, thermomigration phenomena in purely diffusive systems. Temperature variations of the interfacial free energy drive the motion, which occurs via diffusion. We have also considered phase separation phenomena in a temperature gradient to investigate potential anisotropy in domain growth introduced by the gradient.

In (c) we have developed a new method for forward integrating the coupled dynamical equations for a conserved order parameter (at the coarse grained, semimicroscopic level) and hydrodynamic flow fields for two-phase systems in two dimensions using a biharmonic solver. The method has been tested on single drop thermomigration in a temperature gradient, simple multiple drop configurations, coalescence, and phase separation kinetics.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-42

RESPONSIBLE CENTER: LeRC

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*Surfactant-Based Critical Phenomena in Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Eric W. Kaler

University of Delaware

CO-INVESTIGATORS:

M.E. Paulaitis

University of Delaware

**TASK OBJECTIVE:**

Our objective is to characterize by experiment and theory the kinetics of phase separation and the metastable structures formed during phase separation in a microgravity environment. The system we are studying is a mixture of water, nonionic C<sub>12</sub>E<sub>6</sub> ethoxylated alcohol surfactants, and supercritical CO<sub>2</sub> at temperatures and pressures where the coexisting liquid phases have equal densities (isopycnic phases).

**TASK DESCRIPTION:**

1. Locate and characterize ordinary and tricritical points in surfactant/water/SCF mixtures about which two coexisting phases will be matched in density.
2. Examine using scattering methods the non-equilibrium structures inside both the spinodal and binodal regions after quenching with either temperature or pressure jumps through or near a critical point.
3. Examine the kinetics of mixing of two phases "quenched" into one phase.
4. Carry out polymerization in a non-equilibrium phase.

**TASK SIGNIFICANCE:**

The potential ability to control the phase separation of liquid mixtures in a microgravity environment has led us to investigate novel processing strategies for materials synthesis in space. As a cost-effective first step in this research program, we have begun to examine phase separation kinetics and materials synthesis under simulated microgravity conditions in earth-based experiments. These conditions are achieved by using mixtures of surfactants and highly compressible supercritical fluids at elevated pressures to produce two co-existing fluid phases of equal density, thus eliminating the density differences that normally drive phase separation. The driving force for phase separation is obtained by rapid expansion of the supercritical fluid, which can also be polymerized to kinetically capture fluid structures in order to measure their physical, chemical, and mechanical properties. Our goal is to explore different processing strategies and the fabrication of a wide range of materials that would eventually be manufactured in space.

**PROGRESS DURING FY 1994:**

In this report, we describe experiments to locate isopycnic equilibrium phases and to determine "local" phase behavior and critical phenomena for the three-component mixture: water, CO<sub>2</sub>, and the surfactant C<sub>8</sub>E<sub>5</sub>. In addition, we report the results of small angle neutron scattering (SANS) experiments to characterize microstructures that exist at different fluid densities for mixtures of the surfactant C<sub>12</sub>E<sub>6</sub>, CO<sub>2</sub>, and D<sub>2</sub>O, and our progress in constructing a light scattering instrument.

Ternary mixtures of a nonionic C<sub>12</sub>E<sub>6</sub> surfactant, CO<sub>2</sub>, and H<sub>2</sub>O will form three coexisting equilibrium phases at elevated pressures and ambient temperatures. These phases are a H<sub>2</sub>O-rich liquid, a CO<sub>2</sub>-rich gas, and (3) a fluid phase with properties that can be adjusted between those of the other two phases depending on the pressure. In preliminary experiments on mixtures of C<sub>8</sub>E<sub>5</sub>, CO<sub>2</sub>, and H<sub>2</sub>O, we determined that the densities of the H<sub>2</sub>O-rich liquid and the third fluid phase would approach one another with increasing pressure. Based on these experiments, we then predicted the temperatures and pressures at which isopycnic phases would form using an equation of state we

developed for such mixtures. As a result of our calculations, we conducted experiments to search for isopycnic phases for mixtures of  $C_8E_3$  and  $C_8E_3$  with  $CO_2$  and  $H_2O$  at temperatures near  $40^\circ C$  and pressures up to 400 bar. A phase inversion was observed for the  $H_2O$ -rich liquid and the second fluid phase at approximately  $32^\circ C$  and 396 bar, and  $30^\circ C$  and 310 bar. Wetting phenomena (i.e., the formation of large spherical droplets of one phase within another) were also observed at these state points. The densities of the two phase were measured at approximately  $30^\circ C$ , and the density difference was found to decrease with increasing pressure from  $1.8 \times 10^{-3}$  g/cc at approximately 91 bar to  $3.0 \times 10^{-4}$  g/cc at 142 bar. These measurements support our predictions of an isopycnic state point for mixtures of  $C_8E_3$ ,  $CO_2$ , and  $H_2O$ , and suggest that isopycnic phases will exist at accessible pressures and temperatures for mixtures of  $C_8E_3$ ,  $CO_2$ , and  $H_2O$  as well. Our search for these isopycnic state points continues.

SANS measurements were made on mixtures of  $C_{12}E_6$ ,  $CO_2$ , and  $D_2O$  in the water-rich, single-phase region at 7.5 wt %  $C_{12}E_6$  ( $CO_2$ -free basis) in solutions containing different amounts of  $CO_2$ . The distinguishing feature of these measurements is the observed upturn in the neutron scattered intensity, on an absolute scale, as the magnitude of the scattering vector decreases. This feature is usually attributed either to the presence of the attractive interactions between the  $C_{12}E_6$  micelles or to critical scattering. Assuming that particle orientations and interactions are decoupled, it is possible to separate the effect of particle shape (form factor) and interactions (structure factor) on the measured intensity. Given the shape of the curves, a modified Ornstein-Zernike structure factor together with a polydisperse spherical form factor were chosen to model the data. The fits of the model to the data were excellent. An increase in osmotic compressibility with increasing  $CO_2$  concentration is observed which indicates an increase in attractive interactions as  $CO_2$  is added. This observation is consistent with two other experimental observations: a depression in the cloud-point curve with increasing  $CO_2$  concentration and negative excess molar volumes for mixtures of  $H_2O$  and  $CO_2$  with a similar surfactant,  $C_8E_3$ . The lack of any significant change in either polydispersity or size of micelle with increasing  $CO_2$  concentration leads to the conclusion that  $CO_2$  has a minimal effect on colloidal microstructure at these concentrations. This is supported by the variation in the contrast parameter with pressure, temperature, and  $CO_2$  concentration. Using the variation of this parameter, it was determined that little or no  $CO_2$  was entering the micelle, but instead remained in the water where it was below its solubility limit.

An apparatus for high-pressure light scattering has been designed and assembled. The key component of this apparatus is the sample cell, and we have tested first quartz and then sapphire as materials of construction that can withstand high pressures. A preliminary theoretical analysis of scattering has also been undertaken, following the work of Binder and coworkers, who have predicted the time variation of the structure factor for a mixture undergoing phase separation.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-75

RESPONSIBLE CENTER: LeRC

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*Instability of Velocity and Temperature Fields in the Vicinity of a Bubble on a Heated Surface*

PRINCIPAL INVESTIGATOR: Dr. Mohammad Kassemi

Ohio Aerospace Institute

CO-INVESTIGATORS:

Dr. Nasser Rashidnia

NYMA, Inc., NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objective of this combined experimental/numerical work is to investigate the fluid flow and temperature fields in the vicinity of a bubble attached to a heated solid surface. The goal of the research is to investigate the interaction between thermo-capillary and natural convective flows in the vicinity of the bubble and identify numerically the parametric range for which unsteady periodic oscillations occur on earth and in the low-gravity environment. It is hoped that the experience gained during this investigation will aid in designing and proposing the *right* space experiment in the future.

**TASK DESCRIPTION:**

Ground-based experiments are performed to identify the following criteria:

1. the appropriate experimental design and fluids
2. the required diagnostic tools
3. the parametric ranges for which oscillatory flow and temperature fields are obtained.

A numerical model is developed and its accuracy will be tested by ground-based experimental results. The verified numerical model will be used to predict the fluid flow and temperature fields in the low-g environment. Drop tower experiments will be performed to experimentally verify the effects of low-g environment on the temperature and flow fields. The experience and knowledge base gained during this effort will be used to design an experimental system for both 1-g and low-g applications.

**TASK SIGNIFICANCE:**

The oscillatory thermocapillary phenomenon is not only of fundamental importance in understanding the behavior of fluid flow in space, but it additionally has a practical significance in materials processing. For example, in crystal growth, the oscillatory temperature and flow fields created by unavoidable bubbles next to the solid-liquid interface may seriously affect the quality of the emerging crystal. In addition, the results of this investigation will enhance our understanding of the role of capillary forces in fluid management and boiling processes in space.

**PROGRESS DURING FY1994:**

Although the funds for this research were initiated in Fall of 1994, significant progress has already been made. The design and construction of a test cell for preliminary experiments has been initiated. Interesting results have been obtained using this simple test cell. Application of Mach-Zehnder interferometry in 1-g experiments simultaneously with a laser sheet flow visualization technique has enabled the observation of steady and periodic oscillatory motions in the vicinity of the bubble on a heated surface.

As part of the theoretical/numerical study, the equations describing the temperature and fluid flow fields around the bubble were formulated and cast into dimensionless form using appropriate scaling parameters. A finite element numerical model was developed and used to simulate the temperature and fluid fields around the bubble. Steady state temperature and velocity fields predicted by the finite element model are in excellent qualitative agreement with the experimental results. More rigorous quantitative comparisons are underway. A parametric study of the interaction between Marangoni and natural convective flows including conditions pertinent to microgravity space experiments are in progress.



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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/94    EXPIRATION: 12/96

PROJECT IDENTIFICATION: 962-24-05-86

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Presentations**

Rashidnia, N. "Instability of flow and temperature fields in the vicinity of a bubble on a heated surface." presented in the 2nd Microgravity Fluid Physics Conference, NASA Lewis Research Center, Cleveland, OH, 1994.

*Stabilization of Thermocapillary Convection by Means of Nonplanar Flow Oscillations*

PRINCIPAL INVESTIGATOR: Prof. Robert E. Kelly

University of California, Los Angeles

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

It has been demonstrated theoretically that small amplitude, nonplanar flow oscillations can stabilize Rayleigh-Benard convection. The first goal of this research is to discern if similar stabilization can be achieved for the thermocapillary (Marangoni) instability by the same means.

**TASK DESCRIPTION:**

An asymptotic expansion based on Reynolds number will be used to perform stability analysis on both Rayleigh-Benard and Marangoni-Benard convection. A numerical approach using a Fourier representation in the horizontal directions and a spectral approach in the vertical direction will be used to obtain a coupled set of ordinary differential equations in time which can be solved by use of Floquet theory.

**TASK SIGNIFICANCE:**

If stabilization is possible, then the effects of finite amplitude oscillations will be determined in order to see how much stabilization is possible. And if significant stabilization is predicted, an experiment will be proposed at a later time.

**PROGRESS DURING FY 1994:**

During the initial phase of the investigation, an analysis suitable for small amplitude oscillations was carried out. These results have now been accepted for publication (Or and Kelly, 1994). The main conclusion is that the oscillations can be either stabilizing or destabilizing, depending on the deformability of the surface of the fluid layer. For low deformability (i.e., small Crispation and Bond numbers), stabilization is predicted whereas destabilization is predicted for high deformability.

The focus during the current phase has been determining the extent to which stabilization or destabilization can occur for finite amplitude oscillations by means of a numerical analysis. Preliminary results reported at the 2nd Microgravity Fluid Physics Conference indicated that changes of 300-500% in the critical Marangoni number can be realized for moderate values of characteristic Reynolds number (nearly 25), and so the effect should be observed readily in an experiment. The numerical calculations are being done by Dr. Arthur Or.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-24-05-56

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Or, A.C. A shooting scheme for boundary-value problems. J. Comp. Physics A. vol. 3, 280-183 (1994).

Or, A.C. and Kelly, R.E. Onset of marangoni convection in a layer of fluid modulated by a weak nonplanar oscillatory shear. accepted for publication by Int. J. Heat Mass Transfer.

### **Presentations**

Kelly, R.E. "Stabilization of thermocapillary convection by means of nonplanar flow oscillations." Presented at the 2nd Microgravity Fluid Physics Conference, Cleveland, OH, June 1994.

Or. A.C. and Kelly, R.E. "Stabilization of marangoni convection by means of nonplanar shear oscillations." AIAA paper 94-0242, presented at 32nd Aerospace Sciences Meeting, Reno, NV, January 1994.

### *Microgravity Heat Transfer Mechanisms in the Nucleate Pool Boiling and Critical Heat Flux Regimes Using a Novel Array of Microscale Heaters*

PRINCIPAL INVESTIGATOR: Prof. Jungho Kim

University of Denver

Co-INVESTIGATORS:

Cameron Moore

University of Denver

#### **TASK OBJECTIVE:**

The objectives are to determine the relative contribution of the various heat transfer mechanisms to the overall heat flux in a sub-cooled, nucleate pool boiling in a microgravity environment, and to obtain quantitative data measuring local heat transfer coefficients at critical heat flux conditions.

#### **TASK DESCRIPTION:**

A two-dimensional array of microscale heaters will be used for these tests to provide control and make measurements. These heaters will be kept at a constant temperature and their power input will be measured to control the heat flux. With the ability to measure and control temperatures and heat flux over very small areas, it should be possible to adequately quantify some of the behavior during nucleate pool boiling and critical heat flux during various stages of bubble growth. Testing would be limited to normal gravity tests.

#### **TASK SIGNIFICANCE:**

The development of such a microscale heater could significantly enhance the state-of-the-art in scientific instrumentation in the area of flow boiling and two-phase flow. Understanding the boiling phenomena has tremendous impact on electronic cooling and the nuclear, oil, and electric power industries.

#### **PROGRESS DURING FY 1994:**

Based on a proposal submitted to the Annual NRA, a grant was awarded in May 1994. Progress on the project has been made in two main areas: the heater construction, and the electronic hardware.

All of the steps necessary to fabricate the heater array has been performed using some dummy masks, verifying the feasibility of constructing the heater. Work is being performed on identifying an appropriate process by which to construct a resistor with the desired characteristics.

A prototype bridge circuit to be used for the heater feedback loop was constructed and tested using a thermistor (which has a negative temperature coefficient of resistance) as the heating element. With low current, the circuit was able to detect a breeze blowing through a window. With high current, the circuit was able to maintain a constant temperature high enough to boil water. A printed circuit board containing 16 of these circuits per board is currently being laid out, but final design of the circuit must wait until the actual heaters to be used are available. Ten of these cards (for a total of 160 circuits) will be used to control the heater array. Much of the design of another circuit that interfaces between the computer and the feedback circuits has been completed. This circuit will enable computer control of the control resistor values in the feedback loop, which will allow the temperature of the heater array to be changed very easily.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 5/94 EXPIRATION: 5/96

PROJECT IDENTIFICATION: 962-24-00-87

RESPONSIBLE CENTER: LeRC

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*Molecular Dynamics of Fluid-Solid Systems*

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PRINCIPAL INVESTIGATOR: Prof. Joel Koplik

City College of New York

CO-INVESTIGATORS:

J.R. Banavar

Penn State University

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**TASK OBJECTIVE:**

The purpose of this theoretical research is to examine the molecular behavior of fluids in small confinements and near boundaries, using molecular dynamics calculations and the statistical mechanics of classical fluids. It looks at time and spatial scales where continuum fluid mechanics provides no insight.

Examples investigated are: (1) static and flowing pure fluids near walls, (2) freezing transition in small pores, (3) the breaking and coalescence of droplet interfaces driven by gravity of flows, and (4) droplet spreading on solid surfaces.

**TASK DESCRIPTION:**

The numerical calculations are based on Molecular Dynamics, wherein one integrates the (classical) equations of motion for interacting molecules of various species, based on a prescribed interaction and specified thermodynamic operating conditions and mechanical forces. Within the framework of molecular dynamics, it is straightforward to impose a desired value of gravity or simulate the presence of g-jitter.

**TASK SIGNIFICANCE:**

We intend to carry out a number of calculations involving coexisting liquid, vapor and solid phases under flow conditions, with two common features. First, the systems will be studied at the molecular scale to obtain otherwise unavailable information. Second, the problems to be studied deal with fundamental issues in the physics of fluids and interfaces that are of relevance to the microgravity program.

**PROGRESS DURING FY 1994:**

We have carried out molecular dynamic simulations of fluids of simple linear molecules between two parallel atomic plates. Our focus has been on monitoring the behavior of the surface layers in very narrow channels for flowing and for supercooled liquids. While both no-slip and slip behaviors are found, depending on the wall-fluid interaction, the behavior of flows in molecular-sized channels is found to be described by continuum hydrodynamics. While the amorphous surface layers do not make the fluid flow non-Newtonian, they suppress the novel wall-induced mechanism of freezing observed in fluids containing spherical molecules. Indeed, we were able to observe the return of the wall nucleation mechanism on reducing the bond length of the molecules. We have also simulated the stick-slip motion of two atomic plates separated by a lubricant layer made of either spherical or dumbbell molecules, in order to evaluate the changes caused by the additional degrees of freedom of the confined fluid. The simple phenomenon of ordering and disordering of the lubricant associated with stick and slip is supplanted by more complex behavior in the presence of dumbbell molecules.

Studies are underway of effects of a nearby wall on a ball falling in a fluid. In the bulk, Stokes' law relating the force on a solid ball to its velocity is reproduced by the molecular dynamics simulations. Continuum studies, primarily due to Brenner, have focused on the force and torque on a ball as it gets close to a solid surface. Our results indicate excellent agreement with the continuum calculations until the ball gets to within a few molecular diameters from the wall at which point, deviations are observed. The effects of the molecular structure of the wall and the ball, the wetting properties of the wall surface and roughness are currently being probed.

We have studied the small-scale dynamics of the apparently singular flow which arises in the sliding plate problem, a driven cavity flow where discontinuous boundary velocities at corners produce a shear stress singularity. MD simulations indicate that the singularity is smoothed by fluid slip accompanied by non-Newtonian behavior in the corners, although the flow is in good agreement with Navier-Stokes calculations elsewhere. The results are not compatible with a Navier slip boundary condition, however. By consideration of a range of Reynolds numbers, we find that the non-Newtonian behavior is associated with strain rates comparable to the internal frequencies of the molecular motion.

In order to explore the origins and dynamics of hysteresis in spreading processes, we have done MD simulations of a Wilhelmy-plate apparatus, in which a solid plate is drawn through the interface between immiscible fluids. The force vs. velocity characteristics, advancing and receding contact angles, and the velocity and stress fields associated with the contact line motions have been obtained. We have also extended our earlier study of terraced wetting phenomena, by simulating the spreading of drops made of longish chains (lengths 8 and 16) on a substrate. The growth velocity of the layers appears to approach  $\sqrt{t}$  as the molecule length increases, confirming an earlier conjecture on the difference between our original simulations and experiment.

We have written a systematic review of MD simulations of fluid flows, which will appear in next year's Annual Review of Fluid Mechanics.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 4

TASK INITIATION: 6/90 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-43

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**
**Journals**

Kablinski, P., Ma, W.-J., Maritan, A., Koplik, J. and Banavar, J.R. Domain growth near a wall in spinodal decomposition. Phys. Rev. Lett., vol. 72, 3738 (1994).

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Ma, W.-J., Kablinski, P., Maritan, A., Koplik, J., and Banavar, J.R. Dynamical relaxation of the surface tension of miscible phases. Phys. Rev. Lett., vol. 71, 3465 (1993).

### *Fluid Dynamics and Solidification of Metallic Melts (FDSMM)*

PRINCIPAL INVESTIGATOR: Prof. Jean N. Koster

University of Colorado, Boulder

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective of this effort is to investigate surface tension-driven convective flow of low Prandtl number melts, e.g., metals, and their effect on the solid-liquid interface. (The Prandtl number is the ratio of the kinematic viscosity to the thermal diffusivity.) This will require flow visualization which will be achieved by using tracer particles and a real time X-ray system. This research will focus on fluid physics phenomena of surface-tension-driven flows in material processing. It is known that liquids for which  $Pr > 1$  have different underlying fluid physics than liquids for which  $Pr \ll 1$ . The research is focused on gallium melts.

#### TASK DESCRIPTION:

Progress toward achieving radiographic particle image velocimetry (RAPIV) of liquid metal convection has required extensive technological development. The RAPIV will be used to obtain system flow velocity vector fields using appropriate computational analysis. RAPIV has been used successfully to detect the solid-liquid interface of solidifying gallium and to observe tungsten particles in molten gallium.

Rectangular, two-dimensional test cell geometries for the series of high temperature RAPIV experiments have been chosen. Two aspect ratios (length:height) will be used: 4:1 and 1:1 (with unit integration depth). The 4:1 ratio is the classical "Hurler" geometry which is used often for comparative numerical studies.

The Integrated Convection Apparatus and Rotating Undercarriage Support (ICARUS) is a high-temperature (up to 1,000°C) modular furnace capable of establishing any combination of vertical and horizontal gradients in test cells of various geometries. This provides the capability to vector the gravity body force from 0 deg. (horizontal) to 90 deg. (vertical).

#### TASK SIGNIFICANCE:

Fluid physics is the foundation of material solidification. The primary purpose of this research is to develop a unique research capability for convective flow visualization of metallic and electronic melts during the solidification process in real time. By observing the flow pattern of a liquid metal during solidification, knowledge will be gained in understanding the solidification process. This knowledge can be used to build better processing facilities which can produce metals and electronic materials with improved properties (e.g., yield strength and toughness).

For example, titanium is used in today's aircraft, but little is known about titanium solidification. The design engineer uses a safety factor of 1.33 which causes the parts to be heavier than they need to be. If the solidification process was better understood, the titanium parts would be lighter. This would reduce the aircraft weight, thereby improving the fuel efficiency or allowing greater payload capacity.

#### PROGRESS DURING FY 1994:

The advanced research facility for liquid metal ( $Pr \ll 1$ ) flow visualization using an X-ray system and high temperature furnace (up to 1000°C) has been built and tested. Gallium has been selected as the test material. Visualization of the density field has been achieved with the X-ray system. A density change of 0.1% can be detected and is enhanced using false coloring. A 10°C horizontal temperature gradient was imposed. The mottle is obscuring some of the data; this problem is being addressed. Eventually, flow patterns will be observed using tracer particles. The particles must exhibit good wetting characteristics, high X-ray absorption and be neutrally buoyant.



The development of neutrally buoyant, chemically inert tracer particles with high radiographic absorptivity is essential to the RAPIV project. A substantial ongoing portion of the development effort has been directed toward the design of the appropriate tracer particles: The particles are currently under development. The most promising particles are glass particles coated with nickel with a final layer of gold. Testing is in progress.

STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	1
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 2/91 EXPIRATION: 7/95

PROJECT IDENTIFICATION: 962-24-00-33

RESPONSIBLE CENTER: LeRC

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**Journals**

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Prakash, A. and Koster, N.N. Convection in a liquid B203 encapsulated GaAs melt. *Int. Jl. Heat Fluid Flow*, (1994).

*Thermocapillary Convection in Floating Zones under Simulated Reduced Gravity*

PRINCIPAL INVESTIGATOR: Prof. Sindo Kou

University of Wisconsin, Madison

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The main objective of the research is to enable comparison between calculated and observed patterns of thermocapillary convection in floating zones, regardless of whether or not the zones are cylindrical in shape.

Ground-based flow visualization experiments will be conducted under simulated reduced-gravity conditions, i.e., where thermocapillary convection dominates over natural convection just as in microgravity. This will include silicone-oil zones and molten zones in  $\text{NaNO}_3$  rods. Computer simulation of thermocapillary convection will be conducted and the calculated results will be compared with those observed in flow visualization.

**TASK DESCRIPTION:**

A mathematical formulation will be used to quantitatively describe the optical distortions. This formulation allows the calculated flow patterns and solid/liquid interfaces to be converted into distorted ones so that they can be compared directly with those observed in flow visualization to verify the validity of computer simulation. Conversely, it can also be used to convert the observed flow patterns and solid/liquid interfaces into undistorted ones.

**TASK SIGNIFICANCE:**

Comparisons between calculated and observed patterns of thermocapillary convection in floating zones, though significant, have been rare (if performed at all), due to complications from optical distortions caused by the lens effect of the floating zones. With the numerical tool, thermocapillary flows in floating zone configurations can be more deeply understood. The capability of prediction can help the design of process of the active control, thus the quality of the material and medicine will be improved.

**PROGRESS DURING FY 1994:**

1. The velocity field in a silicone oil zone was measured using a laser light-cut technique and an optical chopper.
2. The velocity field in a molten zone in a sodium nitrate rod was measured in the same manner.
3. The velocity fields were calculated by computer simulation.
4. The comparison between the calculated and the observed results was very good. It was demonstrated that without taking into account the lens effect of the floating zones, the calculated and the observed results can differ from each other significantly.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-46

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Journals**

Lan, C.W., and Kou, S. Radial dopant segregation in zero-gravity floating-zone crystal growth. *J. Cryst. Growth*, vol. 132, 578-591 (1993).

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### *Analysis of Phase Distribution Phenomena in Microgravity Environments*

PRINCIPAL INVESTIGATOR: Prof. Richard T. Lahey

Rensselaer Polytechnic Institute

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective of this research is to map the void distribution throughout the tube cross-sectional area in two-phase flows. The principal flow patterns to be studied are the bubbly and slug-flow regimes.

#### TASK DESCRIPTION:

The research approach is to conduct normal gravity testing using equal density simulation experiments. Normal gravity experimental data, as well as available microgravity data, will be used to verify computational fluid dynamic models that are being developed. Two types of equal density simulations will be used: liquid with solid spheres, and a pair of immiscible liquids.

#### TASK SIGNIFICANCE:

The orientation of the phases with respect to each other and the tube wall affects the heat transfer characteristics and the work required to pump the two-phase mixture. Terrestrial applications include the nuclear, electric power and oil industries.

#### PROGRESS DURING FY 1994:

Data acquisition and processing has been completed for testing with a water/polystyrene mixture (liquid - neutral buoyant particles). The radial distributions of the following measurements were obtained: average liquid velocity, average particle velocity, turbulent intensities for the liquid and the particles, and the particle volume fraction. The relative velocity between phases were close to zero, which is in disagreement with the microgravity data published by Dr. Jean Fabre from the Institut de Mecanique des Fluides in Toulouse, France. After Dr. Fabre was contacted, and the data was reviewed, Dr. Fabre acknowledged that the relative velocity was also close to zero for his tests.

Other results are as follows: the turbulence intensities of both phases are about the same. The particle volume fraction has a maximum at the tube centerline and decreases towards the tube wall. This is opposite to the behavior of liquid/vapor mixtures flowing upwards in normal gravity conditions, where the vapor phase accumulates close to the wall and is dangerous for high heat flux diabatic flows. Fortunately, based on these results the vapor is not expected to accumulate near the wall under microgravity conditions.

The three-dimensional, two-fluid conservation equations (i.e., mass, momentum, and energy balances) and the closure laws using ensemble averaging for the liquid - particle mixture have been derived. The model includes a consistent treatment of the interaction between the liquid turbulence and the particles, the collisions among particles and collisions between the particles and the wall. The solution of the conservation laws has been numerically computed using a CFD finite volume technique (Phoenix). The numerical results agree remarkably well with the experiments without the adjustment and any coefficient.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-00-72

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Presentations**

Lahey, R. and Bonetto, F. "Analysis of phase distribution phenomena in microgravity environments." presented at Second Microgravity Fluids Conference in Cleveland, OH. June 21-23, 1994.

*Nonlinear Drop Dynamics and Chaotic Phenomena*

PRINCIPAL INVESTIGATOR: Dr. L. G. Leal

University of California, Santa Barbara

CO-INVESTIGATORS:

E.H. Trinh

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

The general objective of this collaborative experimental and theoretical project is to explore nonlinear phenomena associated with acoustic and/or electrostatic levitation of drops or gas bubbles in air or a viscous fluid. These include both phenomena intrinsic to large amplitude oscillations of shape (or volume, in the case of gas bubbles), and phenomena that are a consequence of the levitation process. In the former category, we include studies of newly discovered coupling between shape modes and volume oscillations for gas bubbles, and oscillations of non-spherical uncharged and charged drops in electric fields, and in combined acoustic and electric fields. When experiments aimed at understanding or exploring these fundamental, intrinsic phenomena are carried out in an acoustic field, which may be either steady or time modulated, there can be strong coupling between the bubble or drop position or shape, and the acoustic levitation force, which can lead to additional nonlinear oscillatory phenomena that can be difficult to separate from the intrinsic phenomena of interest.

**TASK DESCRIPTION:**

Following earlier work based upon small deformation asymptotics, coupled with a nonlinear dynamics analysis of the weakly nonlinear amplitude equations, current theoretical work is focused upon numerical solutions for finite deformations of shape. Our most recent work has considered an inviscid drop (charged or uncharged) in a steady or time-dependent electric field. We utilize the well-known boundary integral technique for this case, but we have also developed both full finite-difference and spectral techniques to solve free boundary problems with viscous effects.

Experimental studies have utilized ground-based levitation systems located at JPL. These include both acoustic levitation for small gas bubbles, and a combination of acoustic and electrostatic levitation for viscous drops in air. The latter is particularly interesting because it allows compensation for the mean deformation associated with the levitation process, so that the mean drop shape is spherical.

**TASK SIGNIFICANCE:**

We have worked on nonlinear dynamical effects due to time-dependent forcing, and/or an initial deformation for both gas bubbles and viscous drops. These problems are important both from a fundamental point of view as a concrete and accessible physical system which exhibits many of the classical nonlinear phenomena, but also because the problems being studied have significant application to phenomena in multiphase flow. The work on gas bubbles has yielded a fundamentally new understanding of forced or free volume oscillations in the sense that we have shown that purely radial oscillations, without change of shape, is an exceptional phenomena. The generic behavior is that there is an exchange of energy between volume (radial) and shape modes, which becomes extremely strong near resonance. Current numerical and experimental work is aimed at understanding large amplitude effects, including conditions that may lead to sufficiently large oscillations of shape that the bubble breaks. Bubble breakup due to time-dependent fluctuations of an isotropic background pressure has not been investigated previously as a mechanism for breakup, insofar as we are aware.

The dynamics of viscous drops in an acoustic or electrostatic levitator was originally studied in the microgravity program of NASA primarily because it was viewed as the basis for containerless materials processing applications. Although this is no longer a major priority, the fundamental problem is still one of considerable interest, especially as a model system for understanding drop dynamics in electric fields for applications ranging from meteorology (raindrops) to ink-jet printers. There is also a growing interest in the possibility of using time-dependent droplet motions as a basis for measurement of material properties of the liquid or interface at extreme conditions of

temperature. Finally, as with the bubble, the oscillating drop offers a convenient laboratory system to continue to improve our understanding of the theory of nonlinear systems.

#### PROGRESS DURING FY 1994:

The experimental effort in FY 94 has concentrated on four separate tasks: (1) the study of large amplitude shape oscillations of ultrasonically levitated drops in a time-varying electric field; (2) the investigation of large amplitude shape oscillations of large bubbles ultrasonically trapped at reduced hydrostatic pressure; (3) the analysis of mode coupling for nonlinear oscillations of drops levitated in air and drops and bubbles levitated in a liquid medium; and (4) the investigation of large amplitude capillary waves on levitated thin liquid shells.

1. Oscillations of non-spherical uncharged and charged drops ultrasonically levitated in air and in the presence of a constant or time varying electric field have been quantitatively investigated. A soft nonlinearity has been measured for the fundamental mode of shape oscillation, sub-harmonic resonant excitation of the fundamental  $l=2$  and the next higher mode  $l=3$  has been discovered, hysteresis in the large amplitude response of the fundamental mode has been documented, and anomalous free decay frequencies for drops driven into the higher resonant modes have been observed.

2. Air bubbles up to 5 mm in diameter have been trapped in 1 g in an ultrasonic standing wave at 22 kHz and at a hydrostatic pressure reduced to about 0.3 atmosphere. Shape oscillations have been excited for various resonant modes described by spherical harmonic expansion. For the water and air combination, shape modes up to  $l=5$  can be excited to macroscopic amplitude. No observation of coupling to the radial mode has been possible due to the cavitation onset in the host liquid due to the high intensity acoustics required for trapping the bubbles in 1 G. An approach for a microgravity experiment to investigate this radial to shape coupling is being developed.

3. Digital video analysis of shape oscillations of drops and bubbles in a liquid has been used to investigate the resonant mode coupling of driven and freely decaying oscillations. For drops, direct coupling between the  $l=6$  and  $l=3$  axisymmetric modes has been observed for ultrasonically driven oscillations. Similar coupling between the axisymmetric  $l=4$  and  $l=2$  non-axisymmetric modes has been documented. Coupling between the first four resonant modes of freely decaying bubbles has been quantitatively measured through spherical harmonics expansion of the observed oscillations.

4. The observation of the optically scattered signal from a levitated liquid shell has been used to characterize the evolution of large amplitude acoustically-induced capillary waves. An interpretation of the power spectrum of the waves has been attempted using the interaction of symmetric and antisymmetric waves on the shell. High speed video observation has revealed a highly turbulent shell surface corresponding to a continuous frequency spectrum prior to the bursting of the shell.

The theoretical effort in FY 94 has been focused on three specific directions:

(1) Application of our numerical boundary-integral code to study the problem of an electrostatically levitated drop in a gravitational field, where both the charge  $Q$  and the electric field strength  $E$  must be nonzero. We are concerned with steady shapes in steady  $E$  fields, oscillation frequencies for perturbed initial shapes as a function of  $E$  and  $Q$ , and the response to time-dependent electric fields, among other issues. This work is currently being prepared for publication.

(2) Re-examination of the problem of instabilities in the position of particles, bubbles or drops due to coupling with the acoustic field in levitation. We have specifically been studying the computation of acoustic pressure forces and fields when a body undergoes large (or at least not small) changes in position or shape. This has led to a new formulation of the instability of position first studied theoretically by Rudnick and Barmatz (J. Acoust. Soc. Am. 87 (1), 81 (1990)). It is also the basis for numerical computations of time-dependent position and/or shape in an acoustic pressure field.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

(3) We have continued to work on numerical simulations of finite amplitude coupling of shape and volume for a gas bubble in steady and time-dependent pressure fields in support of the corresponding experimental studies.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/91 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-24-07-01

RESPONSIBLE CENTER: JPL

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*Oscillatory Cross-Flow Electrophoresis: Application to Production Scale Separations*

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PRINCIPAL INVESTIGATOR: Dr. David T. Leighton

University of Notre Dame

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The task objective primarily consists of an experimental verification of the expected performance of an oscillatory cross-flow binary separation device. This device is designed to use an oscillatory electric field across the narrow gap of a specially designed electrophoresis channel to cause species with different electrophoretic mobilities to have different time / location histories within the gap. This oscillatory motion is coupled with an oscillatory fluid cross-flow to achieve rapid separation of molecules based on their electrophoretic mobilities. The system acts as a semi-permeable barrier, actively transporting molecules with mobilities either greater or lower than some adjustable value across the cell, while rejecting all others. Because the throughput is largely governed by the amplitude of the oscillatory cross-flow, protein throughputs several orders of magnitude greater than those in conventional continuous free-flow electrophoresis devices are theoretically achievable.

**TASK DESCRIPTION:**

Initially, the design for the oscillatory cross-flow binary separation device will be finalized and the device itself constructed. After construction of the device, its performance will be first qualitatively tested using the dyes phenol red and naphthol green B. As the amplitude of the electric field is changed, the dyes will be selectively allowed to pass through the separation cell from one reservoir to another. Subsequently, quantitative measurements of the separation achieved by the device will be made using the proteins cytochrome-C, albumin and thyroglobulin. These readily available proteins can be analyzed using HPLC equipment available to the PI at the Center for Bioengineering and Pollution Control at the University of Notre Dame. Concurrent with the experimental studies, we will also conduct simulations of the device to investigate end effects, finite concentration effects, and the influence of more general electric fields and oscillatory cross-flows. It is hoped that these investigations will allow us to optimize the device and further improve the selectivity and throughput.

**TASK SIGNIFICANCE:**

Because the amplitude of the throughput is largely controlled by the amplitude of the fluid motion and only indirectly by the electrophoretic mobility, the throughput of this device is potentially several orders of magnitude greater than conventional free-flow electrophoresis devices. Experimental verification of the theoretical concepts underlying the proposed device are crucial, however. The research program is designed to 1) provide such verification and 2) to further optimize the system.

**PROGRESS DURING FY 1994:**

The initial funds for this project were only awarded during the final month of FY94, thus progress to date has been limited. Currently we are in the process of selecting a graduate student to work on the project and finalizing the design of the electrophoresis cell to enable construction

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-24-08-16

NASA CONTRACT No.: NAG8-1080

RESPONSIBLE CENTER: MSFC

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*Low Dimensional Models for Thermocapillary Convective Flows in Crystal Growth Processes*

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PRINCIPAL INVESTIGATOR: Prof. A. Liakopoulos

Lehigh University

CO-INVESTIGATORS:

Prof. P.A. Blythe

Lehigh University

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**TASK OBJECTIVE:**

The objective of this work is to construct and validate low dimensional dynamical models of surface tension driven flows that are relevant to crystal growth processes. The models will be used to study process stability and the use of reduced-order models for control schemes.

**TASK DESCRIPTION:**

A data base of 2-D, axisymmetric, and 3-D numerical solutions of the full nonlinear governing equations will be constructed using spectral element methods. These simulations are based on a variation formulation for unsteady viscous flow and an Arbitrary-Lagrangian-Eulerian description. The Karhunen-Loeve, K-L, procedure is used to determine spatial structures of the computed flow and thermal fields. Coupling of the K-L procedure with the method of weighted residuals for the full models will yield a low-dimensional representation of the flow system.

**TASK SIGNIFICANCE:**

Reduced-order models make feasible stability and bifurcation calculations for thermocapillary flows of practical interest. Furthermore, they can provide the basis for designing model predictive controllers for the suppression of instabilities that occur during crystal growth processes in a microgravity environment.

**PROGRESS DURING FY 1994:**

In the first three months, research has been directed toward the following tasks:

1. Collection of data from direct numerical simulations of thermocapillary flows in rectangular cavities. All simulations are time accurate. If a steady (time-independent) solution exists it is found as the steady-state solution of the appropriate initial boundary value problem after all transients die out. In all cases, the flexibility of the free surface is fully incorporated into the mathematical model. Two configurations are under investigation: a) differentially heated open cavities of aspect ratio (width/height)  $A=1,2,4$  and, b) open shallow cavities with spatially periodic temperature distribution imposed on the free surface. In case (b), the governing equations are solved in a computational domain consisting of a single module with periodic boundary conditions on the side boundaries.
2. Development of computational tools needed for the Karhunen - Loeve decomposition (POD) of 2-D and 3-D oscillatory convective flows and the construction of low-dimensional dynamical models.

The POD methodology has been applied successfully to thermal convection flow in a channel with discrete, spatially periodic heaters. A dynamical model consisting of six ordinary differential equations has been derived by using the empirical eigenfunctions as basis functions in a Galerkin expansion. The stability and bifurcation characteristics of the reduced-order dynamical system are under investigation.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-00-88

RESPONSIBLE CENTER: LeRC

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*Absolute and Convective Instability of a Liquid Jet at Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Sung P. Lin

Clarkson University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective is to establish a definite role of capillary, viscous, and inertial forces in the absence of gravity by using the fluid dynamics problem of the stability of a liquid jet as a vehicle. The objective will be achieved by re-examining the known theories that can be verified completely only in microgravity. A wide range of Weber and Reynolds numbers will be studied, and any unexpected phenomena in microgravity that may require a new interpretation of dynamic capillary force will be examined.

**TASK DESCRIPTION:**

The results of the proposed work will provide some benchmark knowledge in fluid dynamics. When the results obtained at microgravity are compared with the known theories for 0-g and with the known experimental results obtained at 1-g, one will be able to unambiguously assess the significance of gravitational and inertial forces relative to the capillary force over a wide range of dynamic flow parameters. The original approach was to design a rig to conduct one-g experiments during the first year (1993). The approach now, however, is to design and fabricate a rig able to conduct both low-g and one-g liquid jet experiments. Numerical modeling and draft rig designs have been accomplished during the first two years. Ground-based low-g experiments will take place during the second year in the LeRC 2.2-Second Drop Tower. Companion one-g experiments with the same rig will be done at the PI's university. Data analysis and flight experiment definition is tentatively planned for the third year.

**TASK SIGNIFICANCE:**

From a practical view point, the knowledge gained on the precise mechanisms of various modes of jet breakup will allow one to improve many existing important industrial processes. These processes include film coatings, combustion of liquid fuel, and formation of various chemical sprays. Improvements of the efficiency of these processes also bring about a drastic reduction in environmental pollution. The knowledge may also be exploited for advanced material processing.

**PROGRESS DURING FY 1994:**

The theoretical work continued in the investigation of absolute and convective instability of a viscous liquid jet emanating into a gas. As previously reported, this analysis was done over a parameter space spanned by the Reynolds, Froude, and Weber numbers; as well as various viscosity, density, and diameter ratios. The work continued to demonstrate that the reduction in gravity tended to enhance the Rayleigh mode of convective instability (i.e., where the liquid jet breaks up into drops of diameter comparable with the jet diameter). Whereas, the Taylor mode of convective instability (i.e., where jet atomization occurs) is retarded at reduced gravity.

Systems modifications continued this fiscal year in the build up of an experiment rig suitable for the LeRC Drop Tower. Special attention was paid in the design of the electronic components. These components include the function generator and the amplifier. The design and construction is on-going; subsequently, one-g tests have not been done yet. They will be accomplished when the Drop Tower rig has been completed so that both one-g and low-g testing can be done by the same set of hardware. Component position determination and functional testing of the hardware is now on-going at Clarkson on a Drop Tower rig that had been sent to them. It is expected that final rig design and build-up, as well as one-g and low-g testing will occur within the early 1995.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-00-47

RESPONSIBLE CENTER: LeRC

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Lin, S.P. and Webb, R.D. A branching liquid jet. *Physics of Fluids*, 6, 2671-2675 (August 1994).

#### Presentations

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*Magnetorheological Fluids in Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Jing Liu

California State University, Long Beach

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

This research investigates experimentally the influence of gravity in the formation of the equilibrium structure of magnetorheological fluids.

**TASK DESCRIPTION:**

Magnetorheological (MR) fluids are colloidal suspensions whose rheological properties can be varied through the application of an external magnetic field. The key to this modification is the structure induced in the particles of the suspension. This work seeks a better understanding of the equilibrium structures and the influence of gravity to its formation, dynamics of MR fluids, and therefore, the interaction mechanism. This effort will start by identifying the fluids equilibrium structure, characterizing it, and then examining the parameters controlling its formation, including the rate of the applied magnetic field and the effect of gravitational settling. The basic experimental techniques for this work will be static laser light scattering and optical microscopic imaging.

**TASK SIGNIFICANCE:**

This research will help us to understand the basic physics of a liquid to a solid phase transition of this so called "smart" material under the application of an external field. It will additionally provide guidelines for making technologically important materials of the magnetorheological fluids in general. For example, this material can be used in automobiles as shock absorbers, clutch controls, robotic joint controls, etc., due to the viscosity change controlled by the field. In order to do so, however, we must understand and design them better.

**PROGRESS DURING FY 1994:**

Martin Hagenbuehle, a postdoc from the University of Konstanz, Germany, was hired to work on this project.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-90

NASA CONTRACT NO.: NAG3-1634

RESPONSIBLE CENTER: LeRC

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**Proceedings**

Liu, J. "Dynamics of field-induced structure in monodisperse magneto-emulsions." *Gordon Conference on Macromolecular and Electrolyte Solutions*, Oxnord, CA, February 6-11, 1994.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Liu, J. "Structure study of monodisperse magneto-emulsions." II Int. Conf. on Static and Dynamic Light Scattering, Fehmer, Germany, February, 1993.

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Liu, J. "Field-induced structure of confined monodisperse ferrofluid emulsions." APS, Pittsburgh, PA, March, 1994.

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Liu, J., Lawrence, E.M., Iey, M.L., Flores, G.A., Bibette, J., and Richard, J. "Field-Induced structure of confined ferrofluid emulsion." Proc. 4th Int. Conv. on Electrorheological fluids, Feldkirch, Austria, July 19-24, 1993.

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*Cross Effects in Microgravity Flows*

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PRINCIPAL INVESTIGATOR: Prof. Sudarshan K. Loyalka

University of Missouri, Columbia

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CO-INVESTIGATORS:

Prof. R.V. Tompson

University of Missouri, Columbia

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**TASK OBJECTIVE:**

The research objectives are to:

1. Solve the Boltzmann and the Wang Chang Uhlenbeck equations to determine the flow rates (mass and heat) and the matrix of the phenomenological coefficients, for arbitrary Knudsen number (ratio of mean free path to characteristic flow dimension), for arbitrary gas (vapor) mixtures, realistic intermolecular and gas-surface interaction potentials, and for small (linear), as well as large (non-linear), gradients.
2. Verify the results by acquiring experimental data in a diffusion cell.
3. Explore applications of the results above to simulations of flows in ampoules.

**TASK DESCRIPTION:**

The experimental apparatus will be designed to test the theoretical results. The classified diffusion two-bulb setup with a connecting capillary will be used with the bulbs held at different temperatures. Results from the new theoretical and experimental understandings will then be used to study flows in specific microgravity experiments through discussions with the NASA scientists and engineers.

**TASK SIGNIFICANCE:**

Film growth by chemical/physical vapor deposition is a process of considerable interest in microgravity experiments. The absence of natural convection should allow better control of the growth processes, but it has been pointed out that in the highly nonisothermal ampoules, thermal slip (creep) can become a matter of significant concern even for Knudsen numbers as small as  $10^{-3}$ . Thus, it is important to understand and control the flows that arise from the molecular (rather than the mere continuum) nature of gases and vapors.

**PROGRESS DURING FY 1994:**

The project personnel have to-date solved numerically the Boltzmann Equation for a monatomic gas for rigid sphere molecules and cylindrical geometry, under noncondensing conditions. All phenomenological coefficients have been computed. Initial computations for realistic potentials (monatomic gas), and the velocity and the creep slip, have been completed. The creep slip is found dependent of the type of gas, and results confirm accuracy of recently reported variational results. The variational technique also has been extended, and it has been shown that the planar flows can be computed very efficiently, for all Knudsen numbers, by use of the Burnett solutions. The diffusion slip and the creep slip also have been computed for monatomic gas mixtures. The two bulb apparatus for isothermal experiments has been designed, built, and tested. Experimental data on two gas mixtures (Ar-He, N<sub>2</sub>-He) at several pressures (1 torr to 200 torr total pressure) and mole ratios have been obtained, and are found in good agreement with the theoretical predictions (in the slip regime). Development of a Computer program to allow simulation of deposition in cross flows in idealized geometries (cylindrical tube) has been successful. Ongoing and planned research includes calculations on mixtures for arbitrary pressures, measurements with noncondensing and condensing species (such as mercurous chloride) under temperature gradients, measurements of momentum accommodation coefficients (that affect the flows) for gas mixtures, generalization of the computer program on deposition in other geometries, and development of ideas for a space based experiment in the project area.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11-92 EXPIRATION: 11-95

PROJECT IDENTIFICATION: 962-24-00-64

RESPONSIBLE CENTER: LeRC

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*Controlling the Mobility of a Fluid Particle in Space by Using Remobilizing Surfactants*

PRINCIPAL INVESTIGATOR: Prof. Charles Maldarelli

City University of New York

CO-INVESTIGATORS:

Prof. Demetrius Papageorgiou

New Jersey Institute of Technology

**TASK OBJECTIVE:**

This research program studies theoretically and experimentally how to use surfactants to control the mobility of the fluid interface of bubbles or drops moving through a continuous liquid phase. The interfacial mobility determines the drag exerted on the fluid particle as it is driven through the continuous phase. By controlling this mobility, the steady translational velocity of the drop can be manipulated independent of the force causing the particle to move, and this control may prove useful in material processing under microgravity which requires the management of thermocapillary driven bubbles and drops.

**TASK DESCRIPTION:**

Surfactant affects the interfacial mobility by creating surface tension gradients which resist the interfacial flow: Surfactant molecules dissolved in either the continuous or drop phase kinetically adsorb from the bulk sublayer adjoining the interface onto the surface. Once adsorbed, the surfactant is convected by the surface flow to the trailing pole of the particle. Accumulation at the back end causes kinetic desorption into the bulk sublayer; this increases the sublayer concentration causing a diffusive flux of surfactant out into the bulk. At the front end, the kinetic adsorption depletes the sublayer, and forces a diffusive flux of surfactant from the bulk to the sublayer at the front end. At steady state, diffusive, kinetic and convective fluxes balance, and a concentration gradient develops on the surface with the trailing pole larger than the leading pole. Since surfactant reduces the interfacial tension in proportion to its surface concentration, the leading pole is at a higher tension than the trailing pole. The leading pole tugs at the trailing pole, and this tangential action opposes the surface flow and hinders the interfacial mobility.

To use this retardation mechanism to manipulate the interfacial mobility, the kinetic and diffusive fluxes which maintain the concentration difference across the fluid particle must be controlled. Diffusive fluxes can be controlled by using bulk concentrations large enough to form surfactant aggregates; these aggregates act as sources of surfactant monomer thereby reducing the diffusion gradients and increasing the interfacial mobility. Kinetic fluxes are controlled by the kinetic exchange coefficient, which is determined by the surfactant structure. The overall goal is to investigate both of these mechanisms.

**TASK SIGNIFICANCE:**

The results can be used to control thermocapillary driven bubble motion, e.g., in a glass melt, and in miscibility gap solidification of two phase composites. In each of these examples there is a need for controlling the thermocapillary migration velocity. In the movement of gas bubbles in a melt, it is desired to have as large migration as possible. In the case of miscibility gap solidification, the object is to reduce the migration velocity as much as possible, so that phase separation does not occur.

**PROGRESS DURING FY 1994:**

The first effort has studied kinetic control; as described below, a creeping flow model has been constructed to describe the effect of the kinetic rate constant on the fluid particle terminal velocity, and the kinetic rate has been measured using the pendant bubble method for a polyethoxy surfactant.

A. Creeping Flow Model: Kinetic absorption from the sublayer of the fluid particle (assumed to be at the bulk concentration) to the surface is described by a Langmuir kinetic rate equation. The interfacial mobility is controlled

by the ratio of the characteristic kinetic rate to the surface convective rate. This ratio is denoted by  $Bi$ . A creeping flow hydrodynamic model has been constructed and solved by a weighted residual technique; the dependence of the terminal velocity on  $Bi$  is given for a gas bubble and different values of the Marangoni number ( $Ma$ ). The results demonstrate that as the kinetic exchange increases relative to the convective rate (larger  $Bi$ ), the surface concentration becomes more uniform, the interfacial tension gradient force is reduced, and the terminal velocity increases. As surfactant exchange becomes very slow, surfactant accumulates in a stagnant cap at the trailing pole, and the largest retardation in the particle mobility is obtained.

**B. Measurement of Surfactant Exchange Kinetics:** To measure the kinetic exchange coefficient, the pendant bubble technique enhanced by video image digitization is used. In this technique, a bubble is formed at the tip of a needle immersed in a surfactant solution, and a video image of the bubble is used to determine the interface contour and the surface tension. Two types of experiments are undertaken. In the first, the pendant bubble is quickly created, and surfactant diffuses towards and adsorbs onto the freshly created interface, lowering the surface tension. The dynamic surface tension relaxation is measured until equilibrium is achieved. In the second, the bubble is first allowed to come to equilibrium, and is then quickly compressed, raising the surface concentration and reducing the tension. Surfactant desorbs off the surface and diffuses back in the bulk, increasing the surface tension. In this case, the dynamic relaxation in surface tension back to the equilibrium value is measured. For each case, a mixed kinetic/diffusive model to describe the surfactant transport is constructed, and a parametric fit of the relaxation data determines the diffusion coefficient and rate constant.

The surfactant studied is a polyethoxylated amphiphile. Dynamic tension relaxation data for clean interface adsorption and re-equilibration for several values of the bulk concentration have been measured and compared with the mixed model simulations. From the parametric fitting, values are established for the diffusion coefficient and the kinetic absorption coefficient. The desorption rate constant to the adsorption constant is equal to  $4.6 \times 10^{-4} \text{ sec}^{-1}$ .

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-90

NASA CONTRACT No.: NAG-1618

RESPONSIBLE CENTER: LeRC

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*Stabilization and Low Frequency Oscillations of Capillary Bridges with Modulated Acoustic Radiation Pressure*

PRINCIPAL INVESTIGATOR: Prof. Philip L. Marston

Washington State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this investigation are as follows:

1. Investigate the response of the liquid capillary bridges to acoustic radiation pressure excitation.
2. Acoustically suppress capillary breakup of long liquid bridges.

**TASK DESCRIPTION:**

Liquid bridges surrounded by another liquid will be studied in a modified Plateau tank apparatus. A candidate fluid for the liquid bridge is a silicone-oil/tetrabromoethane mixture. The surrounding fluid is a water bath. The bridge length and location will be adjusted to investigate the mode coupling of the radiation pressure of the surrounding ultrasonic field. The ultrasonic field will be mapped with a hydrophone. For dynamic studies, the spatial and temporal modulation of the radiation pressure will be used to selectively excite mode resonances. After deactivating the transducers, mode frequency and rate of the free decay will be monitored optically. Static bridge shapes will then be measured with CCD cameras attached to digitizers and a personal computer.

**TASK SIGNIFICANCE:**

The proposed research furthers the understanding of the dynamics of capillary bridges which can lead to improved control of float-zone crystal growth processes. In addition, the research may also lead to an increase in dynamic gravity tolerance levels for liquid bridge related phenomena on the Shuttle.

**PROGRESS DURING FY 1994:**

A system has been built for the deployment of neutrally buoyant bridges of a silicone-oil/tetrabromoethane mixture in an ultrasonically excited water bath. The equilibrium diameter of the bridge is fixed by the support but the bridge length (and volume) may be adjusted. The amplitude of the ultrasonic field is modulated at low frequencies representative of the natural frequencies of the bridge. By optically monitoring the response of the bridge while scanning the modulation frequency and adjusting the bridge location, the desired ultrasonic excitation of bridge modes has been demonstrated. Ultrasonic deformation of static bridges has also been observed. Additional studies are underway to determine the natural frequencies as a function of bridge length for weakly damped modes. The mode shapes and radiation pressure coupling are also being investigated.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-91

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Marston, P.L., Trinh, E.H., Depew, J. and Asaki, T.J. Response of bubbles to ultrasonic radiation pressure: dynamics in low gravity and shape oscillations. Dynamics and Interface Phenomena. J.R. Bale et al. (eds) (Kluwer, Dordrecht), 343-353 (1994).

#### NASA Tech Briefs

Marston, P.L., Trinh, E.H., Depew, J. and Asaki, T.J. Oscillatory dynamics of single bubbles and agglomeration in a sound field in microgravity, in Joint Launch + One Year Science Review of USML-1 and USMP-1 with the Microgravity Measurement Group, edited by N. Ramachandran, D.O. Frazier, S.L. Lehoczky, and C.R. Baugher (NASA Conference Publication 3171, Marshall Space Flight Center, AL. NASA Tech Brief, Vol 2, 673-690 (1994).

*Study of Disturbances in Fluid-Fluid Flows in Open and Closed Systems*

PRINCIPAL INVESTIGATOR: Prof. Mark J. McCready

University of Notre Dame

CO-INVESTIGATORS:

Prof. H-C Chang  
Prof. D. Leighton

University of Notre Dame  
University of Notre Dame

**TASK OBJECTIVE:**

The objective of this effort is to examine the different instabilities that can affect a liquid film during gas-liquid flow.

**TASK DESCRIPTION:**

A generalized approach is being undertaken for gas-liquid flow in an open channel with finite length, with or without gravitational stabilization, as well as for the analytically simpler case of liquid-liquid rotating flows. Either gas-liquid low-gravity testing or comparison with existing low gravity data is planned.

**TASK SIGNIFICANCE:**

The presence of waves can enhance the heat transfer between phases but increase the energy required to pump the phases through a pipe network. Terrestrial applications include the nuclear, electric power and oil industries.

**PROGRESS DURING FY 1994:**

The rotating matched-density liquid experiment has been run for a range of conditions where waves are present at the interface. The observed onset of waves agrees well with linear theory. Measurements of steady wave amplitudes agree very well with predictions from the theory derived from a weakly-nonlinear simplification of the complete Navier-Stokes equations and boundary conditions. This appears to be the first time that experimental verification has been provided for finite amplitude theory for an interfacial system. The wave shapes match almost exactly; and the speeds, which differ slightly from linear theory, also agree with nonlinear theory. Nonlinear theory indicates that the primary mechanism of stabilization of the unstable fundamental interfacial mode is by a quadratic interaction with the interfacial overtone and internal modes. This occurs even though the system is not resonant. Surprisingly, Couette flow differs from channel flow where a cubic self-interaction is dominant. The physical reasons for this are being investigated.

Calculated wave shapes for a sheared gas-liquid flow reveal that if gravity is absent, the amplitude and speed are greatly increased. Because atomization in annular flows occurs from the crests of large waves, these results suggest that atomization in microgravity studies should be significantly greater than Earth studies. Hopefully, these theoretical results can be verified by using existing interface tracing data from aircraft studies.

STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	3	PhD Degrees:	0

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-05-55

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Presentations**

Gallagher, C., Kropnewicki, T., Leighton, D. and McCready, M. "Two-layer viscous instability in a rotating couette device." Presented at Second Microgravity Fluids Conference in Cleveland, Ohio, June 21-23, 1994.

*Study of Forced Convection Nucleate Boiling in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Herman Merte, Jr.

University of Michigan

CO-INVESTIGATORS:

Prof. R.B. Keller

University of Michigan

**TASK OBJECTIVE:**

This task is a continuation of previous experimental work examining the effects of buoyancy on forced convection nucleate boiling. In nucleate pool boiling, bubble detachment in normal Earth gravity is usually the result of buoyant forces; in the absence of buoyancy the bubbles have a tendency to remain in the vicinity of the heating surface, eventually causing dryout at the heating surface which results in reduced heat transfer. For boiling in a flowing liquid, an additional mechanism of bubble detachment can occur: detachment due to lift and drag on the bubble, induced by the flow field. Understanding when this mechanism will dominate, and the effectiveness of boiling in such circumstances, is important in making accurate predictions of microgravity boiling behavior. This information will also increase the understanding of boiling in cases where gravity is present.

**TASK DESCRIPTION:**

A closed-loop flow boiling system, using R-113 as the test fluid, has been developed as part of a previous program. The temperature and pressure of the subcooled liquid R-113 are rigorously controlled at the entry to the test section. A portion of one wall of the rectangular test section is electrically heated to provide either a constant temperature or a constant heat flux. Surface temperature and heat flux measurements are made and the bubble growth/departure process is recorded visually. The relative effects of buoyancy are examined by rotating the entire flow loop. Other variables in the experiment are the flow velocity, the power input to the heater, and the amount of liquid subcooling. A long-range goal of the effort is to adapt the current test loop for testing aboard NASA's low gravity research aircraft.

The project has focused upon two general areas of study: obtaining heat transfer coefficients for nucleate boiling over a range of variables, and studying the conditions where dryout (departure from nucleate boiling) occurs. Analysis has been performed in both areas of study to develop physical explanations for the observed results.

**TASK SIGNIFICANCE:**

An understanding of the fundamentals of boiling in microgravity is necessary in order to predict the performance and limitations of space systems where boiling occurs, such as those for thermal control and power generation. Near absence of natural convection in microgravity also provides researchers the opportunity to study fundamental aspects of boiling which can improve performance of terrestrial power generation and process equipment.

**PROGRESS DURING FY 1994:**

A study of the effects of orientation on forced convection flow boiling has been completed. Forced convection-dominated and buoyancy-dominated conditions were observed and a parameter, the two-phase Richardson number, ratio of the buoyant force to inertia forces in single phase flow was identified as the relevant indicator of the dominant regime.

Modifications to the test facility, to extend the range of test conditions that can be achieved, are proceeding:

1. A new pump and flow meter has been installed, which doubles the liquid flow rate in the loop. It is digitally controlled.
2. The temperature and pressure control systems has been changed from analog to digital control; the new system is more precise and easier to operate, and will be better-suited for an aircraft experiment.

3. A new cooling system, to replace the existing tap water system, has been designed and will be built. Besides being suitable for aircraft operation, the new system will also increase the range of subcoolings that can be achieved.

A new experimental effort is underway to examine the effect of heater length-to-width ratio on boiling heat transfer rates and on the departure from nucleate boiling.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	2
PhD Students:	0	PhD Degrees:	0

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TASK INITIATION: 11/91    EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-24-05-68

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**
**Journals**

Brusstar, M.J. and Merte, H. Jr. "The effects of buoyancy on the critical heat flux in forced convection". AIAA J. of Thermophysics and Heat Transfer, 8.2, 322-328 (April-June 1994).

Kirk, K.M. and Merte, H. Jr. Low velocity subcooled nucleate flow boiling at various orientations. Accepted for publication in ASME J. of Heat Trans. (1994).

**NASA Tech Briefs**

Merte, H. Jr. "A study of forced convection nucleate boiling in microgravity", Technical presentation at Microgravity Fluids Physics PI Meeting at NASA Lewis Research Center, Cleveland, Ohio. NASA Tech Brief, (June 15, 1993).

Merte, H. Jr. "Pool and flow boiling in variable gravity and microgravity", Technical presentation at NASA sponsored Second Microgravity Fluid Physics Conference, Cleveland, Ohio. NASA Tech Brief, (June 21 - 23, 1994).

**Presentations**

Kirk, K.M. and Merte, H. Jr. "A mixed natural/forced convection nucleate boiling heat transfer criteria." Heat Transfer 1994, vol 7, 478-484. Proceedings of the 10th International Heat Transfer Conference, Brighton, England, August 14-18, 1994.

Li, L. and Merte, H. Jr. "Bubble dynamics and forced convection boiling heat transfer with low velocity." Report NASA Grant 2-1310. Report No. UM-MEAM-93-09. The University of Michigan, Department of Mechanical Engineering and Applied Mechanics, October, 1993.

*Control of Oscillatory Thermocapillary Convection in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. G. P. Neitzel

Georgia Institute of Technology

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective is to experimentally investigate active control of oscillatory thermocapillary convection in planar layers. The investigative strategy is as follows:

1. Establish the appropriate basic state for a "thin" rectangular flow geometry described in the subject proposal.
2. Establish and detect the oscillatory thermocapillary convection in the form of hydrothermal waves.
3. Suppression and control of the hydrothermal waves.

**TASK DESCRIPTION:**

The basic state will be investigated using available computer programs to help establish the experimental design parameters and boundary conditions. After choosing the relevant design parameters and boundary conditions, the dish which will contain the desired basic state will be constructed. The desired basic state is the return flow basic state of Smith and Davis. LDV will be used to observe and measure the basic state obtained in the experiment. The surface temperature perturbations of the hydrothermal waves will then be characterized either numerically or experimentally. Control or suppression of the hydrothermal waves will be attempted using a CO<sub>2</sub> laser as the heat source. Locations along the free surface that appear to most effectively cancel or suppress the hydrothermal waves will be heated with a laser pulse and the duration and time of pulsing will also be compared with the predicted estimates.

**TASK SIGNIFICANCE:**

Crystals grown by the float-zone process can be improved by successfully suppressing undesirable fluid motion during the crystal growth process.

**PROGRESS DURING FY 1994:**

Recent progress has been made using the apparatus modified following the preliminary experiments. For some layer depths, an instability of the planar basic state in the form of a steady, multicellular structure was detected. It was subsequently learned that when driven to higher Marangoni numbers these states became oscillatory. This was originally detected through the use of flow visualization with both sheet-illuminated particle photographs and a newly implemented shadowgraphic technique. The shadowgraphs appear to be more sensitive than the particle video in detecting the onset of oscillations.

By varying the depth of the layer, we changed one of the principal control parameters, the dynamic Bond number. Additional experiments yielded the exciting result that, for small layer depths (small Bond number), a direct transition from steady, unicellular motion to oscillatory motion was observed. This is the transition expected on the basis of the Smith & Davis (S&D) theory which motivated this project; since the S&D theory assumed zero body force, corresponding to zero Bond number, it was anticipated that we had, indeed, detected hydrothermal waves.

This was pursued with increased vigor following the acquisition of our infrared (TR) camera system, which allowed visualization of the free-surface temperature. The steady, multicellular state shows a pattern of transverse rolls, i.e., rolls with their axes aligned normal to the free-surface velocity. When this mode became time-dependent, the



oscillations progressed from the cold wall as a pair of oblique waves superposed on the transverse rolls. This would be a difficult mode to attempt to suppress. For the low-Bond-number transition, however, a single set of oblique waves was observed moving from the cold wall to the hot one. This again, is in line with the expectations from the theory of S&D and the subsequent nonlinear analysis by Smith. It is also the structure hoped for from the standpoint of control.

We are presently extracting free-surface temperatures from the IR camera data. This will allow the determination of the free-surface temperature gradient in the core of the layer, away from the boundary layers at the heated-cooled walls. This, in turn, will allow the computation of a Marangoni number which is device independent and in agreement with theoretical practice. When this is accomplished, it is hoped that the experimental results, for the low-Bond-number transition sequence, will be in much closer agreement with theory.

We shall begin shortly with implementing the proposed control strategy. Given what we have learned about the influence of buoyancy, we will focus on the oscillatory states which exist at low Bond number. We will most likely restrict the bulk of our efforts to studying the 1 cS silicone oil with which we have had the greatest success. While we would like to investigate a 0.65 cS oil, to allow investigation of the effects of Prandtl number, we have had evaporation problems which render the data suspect.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-24-05-61

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

Riley, R.J. and Neitzel, G.P. "Hydrothermal-wave and steady, multicellular instability of a thermocapillary-buoyancy driven flow." Presented at the 47th Annual Meeting of the Division of Fluid Dynamics of the American Physical Society, Atlanta, GA, November 20-22, 1994.

*Industrial Processes*

PRINCIPAL INVESTIGATOR: Prof. Simon Ostrach

Case Western Reserve University

CO-INVESTIGATORS:

Prof. Y. Kamotani

Case Western Reserve University

**TASK OBJECTIVE:**

The objectives of this study are to gain an understanding of the role of gravity in various industrial processes and to develop new applications of microgravity.

**TASK DESCRIPTION:**

The commercial processes and related basic processes which have been investigated or are being investigated, include supercritical extraction processes, coating flows, formation of bubbles in liquid flow, dynamics of liquid-gas interfaces, transport phenomena in zeolite growth, rotating electrochemical systems, and transport phenomena in crystal growth. The roles of gravity in those processes are being studied by using experimental, numerical, and analytical techniques and potential benefits of microgravity are being assessed. Based on those studies microgravity experiments will be proposed.

**TASK SIGNIFICANCE:**

The roles of gravity in terrestrial and space-based industrial processes are being studied by using experimental, numerical, and analytical techniques, and the potential benefits of microgravity applications of these processes are being assessed. Based on these studies, microgravity experiments will be proposed which will lead to an understanding of how to best utilize the environment of space for various industrial applications.

**PROGRESS DURING FY 1994:**

1. Coating Flows

An experiment on dip coating is being conducted to study the conditions for the appearance of wavy films. The experiment covers the capillary number up to 20 and Reynolds number up to 28. When the non-dimensional film thickness exceeds a certain critical value, the film interface becomes wavy. The nature of the interfacial instability is investigated by flow visualization. According to the present experiment a coating film becomes unstable after certain Reynolds number and Stokes number are exceeded. The applicator geometry also influences the film profile and thickness. One conclusion from the present experiment is that gravity significantly influences the stability of the coating film and thus coating in microgravity is advantageous in obtaining uniform films.

2. Bubble Generation in Microgravity

The main idea in this work is to study single bubble generation with transition from the bubbly to the slug flow regime in a continuous liquid flow under microgravity. We are using the Fluid Flow Diagnostics Loop Facility at NASA/Lewis to run experiments under normal gravity conditions. Both co-flow and counter-flow systems have been designed and are now in the manufacturing process. The 1.27 cm I.D. flow conduit section is assembled and certain modification are made in the data acquisition system. After some testings with the 1.27 cm flow conduit system, pipe diameters and injection nozzle diameters will be varied to observe the changes in flow regime transition.

3. Zeolite Growth

We have found a way to grow large zeolite crystals. By adding nutrients during the growth period it is possible to put the extra nutrients into the existing crystals without increasing the number of crystals, thereby increasing their

average size. A patent has been applied for the method. The time to add the nutrients is determined by observing the shrinkage of the gel mixture. The method is considered to be very effective in growing large crystals in microgravity. However, in microgravity the gel does not shrink, but it becomes dilute gradually as the crystal growth proceeds. We found that by putting a He-Ne laser through the gel it is possible to know structural changes in the gel, which makes it possible to identify the time to add the nutrients. We are also studying theoretically the diffusion processes taking place during zeolite growth in microgravity.

#### 4. Rotating Electrochemical Systems

The mass transfer process in shallow rotating electrochemical cells is being investigated numerically, analytically, and experimentally. Various cases relevant to rotating battery applications (Schmidt number about 3,000) have been computed numerically. When the rotational speed exceeds a certain value, secondary cells appear in the core region. We are also performing a scaling analysis to identify new dimensionless parameters and a mass transfer correlation which can classify the entire range of our interest. Based on the numerical analysis we are constructing a new electrochemical test cell. Sectioned electrodes which are specially designed to measure the local mass transfer rate have been fabricated.

#### 5. Natural Convection in Circular Cylinders

Oscillatory natural convection in circular cylinders filled with a liquid metal is being investigated experimentally. The conditions for the onset of oscillations are being investigated under various conditions. The problem is very complex and many factors influence the oscillation phenomenon (e.g. heating rate, wall thickness).

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	3
PhD Students:	4	PhD Degrees:	3

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TASK INITIATION: 1/89 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-24-05-30

RESPONSIBLE CENTER: LeRC

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#### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

##### Journals

Kamotani, Y., Weng, F-B, Ostrach, S. and Platt, J. Oscillatory natural convection of a liquid metal in circular cylinders. Journal of Heat Transfer, Vol.116, (1994).

Kim, I., Kamotani, Y. and Ostrach, S. Modeling bubble and drop formation in flowing liquids in microgravity. AIChE Journal, Vol.40.No.1, (1994).

Zhang, H., Kamotani, Y. and Ostrach, S. Modeling of the zeolite crystal growth process in microgravity and comparison with spaceflight results. AIAA Paper 94-1796.

##### Presentations

Zhang, H. Kamotani, Y. and Ostrach, S. "Modeling of the zeolite crystal growth process in microgravity and comparison with spaceflight results." 1994 AIAA Aerospace Sciences Meeting, Reno, NV, January 1994.

*Marangoni Effects on the Bubble Dynamics in a Pressure Driven Flow*

PRINCIPAL INVESTIGATOR: Prof. Chang-Won Park

University of Florida

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Perform theoretical and experimental research to investigate the effect of surface contamination on the relative motion of air bubbles in a liquid within a Hele-Shaw (HS) cell. Results of this effort will be used to enhance the understanding of the fundamental physical processes and the importance of interfacial flows that occur in a broad variety of industrial and process of applications.

**TASK DESCRIPTION:**

- Focus the theoretical investigation on establishing a predictive model for the influence of surfactant on the bubble dynamics (especially on the translational bubble velocity in a pressure driven flow).
- Experimentally confirm the theoretical predictions and prove that many of the perplexing bubble shapes are the results of the surfactant influence.

**TASK SIGNIFICANCE:**

The results of this research will provide an understanding of the bubble motion on diffusional flux of heat and mass at the bubble-liquid interfaces. This has a close relationship to a number of applications in direct contact mass and heat transfer devices, bioengineering (e.g., blood oxygenation), and it has direct implication for the fluid systems in space-based applications.

**PROGRESS DURING FY 1994:**

This project was initiated on June 29, 1994. The main objective of this research is to investigate the surfactant-induced Marangoni effects on the motion of bubble/drops in a confined geometry. A Hele-Shaw cell, which is a thin gap between two closely space flat plates, is used as a model system for this study. Preliminary experiments have been conducted to date on the motion of an air bubble and a water drop through a silicone oil. An order of magnitude difference in mobility has been observed between the air bubble and a water drop. This difference is presumably due to the influence of surfactant present in the system as contaminants. A systematic experiment is planned to delineate the surfactant influence.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-92

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Park, C-W., Maruvada, S.R.K., and Yoon, D-Y. The influence of surfactant on the bubble motion in Hele-Shaw cells. Phys. Fluids, Vol. 6, no. 10, 3267-3275 (1994).

*Two-Phase Interfaces in Weak External Fields.*

PRINCIPAL INVESTIGATOR: Prof. Jerome K. Percus

New York University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

This endeavor expects to establish a theoretical framework that describes the interfacial response of fluids that have attractive and repulsive contributions, in interparticle interactions, to external fields — particularly a gravitational field. The most interesting result will be the elucidation of how gravitational forces at the microscopic level play a key role in interface dynamics.

**TASK DESCRIPTION:**

1. Analytical Statistical Mechanical formalisms will be employed. Classical Hamiltonians of the interfaces of interacting fluids will be modified or developed as needed.
2. Gravitational external fields are included, but the formalism will be more general.
3. Equilibrium followed by non-equilibrium descriptions are to be developed. It is expected customary as well as new analytical strategies will be utilized.

**TASK SIGNIFICANCE:**

The basic fundamentals of this project is to explore the statistical mechanical basis of three-dimensional liquid-vapor interface formation when an external field of arbitrary magnitude (such as low gravity) is applied. Such fundamental work is expected to point to new experimental observables which have not been anticipated to date. Much of the statistical mechanical interface work in the past has been limited by available tractable methods and ad hoc postulation of interface existence. Only when observed phenomena such as interface existence and shape are natural outcomes of first principles formalism, can one expect to predict new phenomena from the same formalism that increases our understanding. An example from this work is the prediction of a new phase transition that occurs between the uniform vapor state and one with small droplets.

**PROGRESS DURING FY 1994:**

The following describes three important phases of this project:

1. The existence and shape of an equilibrium liquid-vapor interface has been derived from a formalism where the short range repulsive part and long range attractive part of the intermolecular potential have been treated with separate limits (hydrostatic and mean field, respectively) in the ensemble averages. This work naturally predicts existence and shape, plus a likely observable phase transition not previously known. This work has been accepted for publication in the Journal of Statistical Physics.
2. A second part of the work done in FY94 has dealt with the decomposition of equilibrium states into coarse-grained virtual configurations. It is expected that this will aid in the understanding of the evolution of shapes in arrays of bubbles and droplets, and in foams. A report has been submitted to the Journal of Statistical Physics.
3. The third part of this project moves to nonequilibrium dynamics. Nonequilibrium Monte Carlo techniques are being used to predict cluster growth rates and test analytical models. Also, interfacial motion is being studied by the use of multiple time scale separation growing out of the multi-configuration decomposition referred to earlier. The latter work will be a focus for FY95.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-76

NASA CONTRACT NO.: NAG3-1414

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Keissling, M.K.-M. and Percus, J.K. Non-unification Vander Waals theory. Journal of Statistical Physics. (accepted).

*Containerless Capillary Wave Turbulence*

PRINCIPAL INVESTIGATOR: Dr. Seth J. Putterman

University of California, Los Angeles

CO-INVESTIGATORS:

M.B. Barmatz

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

We are working toward the goal of studying turbulence in a broad-band spectrum of capillary waves that run around the surface of a containerlessly positioned drop of liquid. This experiment would constitute the first controlled measurement of turbulence in interacting waves.

**TASK DESCRIPTION:**

The problem consists of two components. They are (a) the generation of a turbulent distribution of surface ripples and (b) the detection and measurement of this state. These issues are being approached in ground-based experiments as well as in arrangements that simulate containerless fluids in microgravity. The ground-based experiments are being carried out in a fluid which is excited with a shake table. The preflight experiments are being developed with a levitated droplet of liquid.

**TASK SIGNIFICANCE:**

Consequences of this experiment range from the characterization of turbulence to the determination of universal properties of nonlinear systems and signal processing. The presence of a new propagating mode (second sound) in the capillary turbulence would have important ramifications with regard to attempts to achieve controlled thermonuclear fusion.

**PROGRESS DURING FY 1994:**

We have achieved the first controlled observations of the transition to broad turbulence in nonlinear wave interactions. These experiments studied the motion of ripples propagating on the surface of water. The key to these measurements is our ability to determine the height of the rippled off-equilibrium fluid as a function of location and time. The fluid is set in motion with a vibration exciter ("shake table"). A CCD camera mounted above the liquid and focused on the surface records the intensity of light that leaves the surface. By doping the water with "polyballs" (neutrally buoyant spheres with 1 micrometer radius) the light is forced to diffuse through the water, so that the intensity at the top surface is proportional to its local elevation. This new technique (diffusive light imaging) is our key advance. It circumvents the catastrophic problems posed by caustics in the former shadowgraph imaging technique.

Our technique can be used to measure the power spectrum or the instantaneous surface topography. Thus the first records of the full 3D profile of a localized soliton state has been obtained. Our data shows a power spectrum of surface vibration given approximately by  $\xi^2(\omega) \sim \omega^{-4.6}$  where  $\xi(x,y,t)$  is the surface height and " $\omega$ " is the frequency. This compares with a theoretical dependence of  $\omega^{-17/6}$ .

Whether we have found a mistake in the theory or if the effects of boundaries (e.g. the side walls of the dish) and a limited dynamic range has led to this discrepancy will be best answered by a flight experiment!

In a space flight the ripples will be excited on the surface of a levitated drop. For this reason the JPL group has been studying the dynamics of large amplitude ripples on a levitated drop of fluid. Excitation of large amplitude waves has been achieved with a "sting" and through modulations of the ultrasonic levitating field. Displacement spectra of the surface oscillations of levitated drops have been obtained using a modified Polytec PI interferometric laser vibrometer with a front-end anti-slosh optics developed by NASA Lewis (Meyer/Tin/Taylor/Mann) for

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

UCLA/JPL (Putterman/Barmatz/Biswas). This front end optics design was also applied to measure displacement power spectrum on a flat liquid surface with amplitudes as high as seven hundred microns (well into the turbulence regime). This new instrument is under development at NASA Lewis (funded by Code UG) and will be assigned to JPL to assist in characterizing surface capillary wave phenomena on a levitated drop.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 12/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 963-24-07-03

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Greenfield, A., S. Putterman, W. Wright. Observation of a localized chaotic structure in a non-linear oscillatory lattice. Physics Letters, (To be published).



*Studies of Radiation-Driven and Buoyancy-Driven Fluid Flows and Transport*

PRINCIPAL INVESTIGATOR: Prof. Paul D. Ronney

University of Southern California

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The proposed research will consist of two sets of experiments under simulated microgravity condition. The first is a study of instabilities of radiating, initially-homogeneous gas volumes, for both optically-thin and optically-thick gases with vertical temperature gradient between two plates. The objectives are to determine the validity of theoretical stability criteria and to determine the spectrum of the nonlinear evolution of the resulting densifications. The second set of experiments is a study of the stability of one-dimensional steady temperature profiles and unsteady thermal conduction waves in gases with strongly temperature-dependent radiative conductivity. The objective is to determine the stability criterion, study nonlinear evolution's of these instabilities, and develop a stability model.

**TASK DESCRIPTION:**

The following are proposed problem studies:

1. Stability of a cooling optically-thin gas volume (field instability) using  $\text{NH}_3$  gas condensible  $\text{I}_2$  vapor or other gas with analogous properties, and measurement of the power spectrum of any resulting nonuniformities.
2. Stability of a cooling optically-thick gas volume which is opaque only at high temperatures (CMBR instability) using CO or other gas with analogous properties, and measurement of the power spectrum of any resulting nonuniformities).
3. Radiative conduction in optically-thick gases between parallel plates using  $\text{SF}_6$ .
  - a. Radiatively-induced instabilities of steady planar conduction profiles at  $\mu\text{g}$ .
  - b. Unsteady planar conduction at constant heat flux (thermal waves) at one-g and  $\mu\text{g}$ .
  - c. Critical Rayleigh numbers at one-g at large  $t$  and  $\Delta T$ .
4. Radiative conduction in optically-thick gases at  $\mu\text{g}$  in spherical geometry.
5. Theoretical stability corresponding to items 3 and 4

**TASK SIGNIFICANCE:**

The study of the coupling of internal radiation to fluid flow is important in a wide variety of practical problems including glass and semiconductor processing; oceanographic, atmospheric and astrophysical flows; plasma physics; combustion systems; solar energy collection; and heat transfer in inhabited enclosures. This work consists of a series of experiments to be carried out in the drop towers that will enhance the understanding of the physics of internal radiation to fluid flow. Results obtained from the present research could identify potential mechanisms to improve the Earth's environment and improve solar power systems.

**PROGRESS DURING FY 1994:**

We have presently constructed an apparatus consisting of two parallel flat plates, each 12 cm x 12 cm, with a gap between the two varying from 2 cm to 5 cm. The upper plate will be heated to an adjustable temperature using an electrical heater imbedded in the surface facing away from the lower plate. The lower plate will be maintained at a constant temperature (approximately 300 K) by ten thermoelectric cooling devices embedded in the plate in a manner similar to that of the upper plate. The plates have been constructed of aluminum so that their thermal response

time will be very large compared to the low-gravity test duration. A well insulated, sealed aluminum chamber to contain the plates and the radiating gas.

We have constructed a shearing interferometer system for flow visualization, since the fluid flow will not be visible to the human eye. This particular interferometer is advantageous because we can extract both qualitative and quantitative information from the images produced and because it is more robust than other interferometer systems. To record the visual images, a camera will be mounted to the structure. It will be linked to a recording device on the drop platform by a sufficiently long fiberoptic cable, provided by the drop tower facility.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-93

RESPONSIBLE CENTER: LeRC

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*Fluid Creep Effects on Near-Wall Solute Transport for Non-Isothermal Ampoules and Suspended Particle Transport Coefficients*

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PRINCIPAL INVESTIGATOR: Prof. Daniel E. Rosner

Yale University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

This experiment focuses on the formulation of fluid creep (slip)/solid boundary conditions over a broad range of fluid densities. This proposal seeks to initiate the theoretical studies necessary to provide the basis for more realistic ampoule-level numerical simulations for vapor transport, including supercritical vapors, and to provide predictions of particle transport properties based on similar phenomena occurring at the particle level.

**TASK DESCRIPTION:**

The following are issues to be addressed in this investigation:

1. Slip coefficients for polyatomic gases, including nondilute, disparate molecular weight mixtures.
2. Simultaneous effects of solid wall creep due to solute mass-transfer, including the coupling between creep associated with energy and mass transfer.
3. Nature of the transition to that of liquid-like densities.
4. Appropriate "creep" conditions at "porous" solid surfaces and implications for the transport properties of suspended particles.

**TASK SIGNIFICANCE:**

Knowledge of these phenomena will allow a greater understanding of the crystal growth process which affects a number of industries (including pharmaceutical/medical).

**PROGRESS DURING FY 1994:**

This grant was funded on July 17, 1994. The appropriate personnel are being brought on board and equipment is being purchased.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 2

TASK INITIATION: 6/94 EXPIRATION: 5/96

PROJECT IDENTIFICATION: 962-24-05-94

RESPONSIBLE CENTER: LeRC

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### *Dynamics of Superfluid Helium in Low Gravity*

PRINCIPAL INVESTIGATOR: Dr. Graham Ross

Lockheed Missiles & Space Co.

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

The objective of this project is to simultaneously record the 3-axis acceleration time history and make a video recording of the position of superfluid helium (SFHe) in a test cell while in a low-gravity environment. This data can then be used as a benchmark for validation of a 3-D CFD simulation of SFHe flow behavior.

#### TASK DESCRIPTION:

The first part of the project is to build a SFHe dewar and a support equipment package to allow operation of the dewar and recording of 3-axis accelerations and video images of the fluid motion on the KC-135. The dewar will have optical windows in the side to allow viewing of the liquid helium in the inner test cell. The data recording is intended to be digital data stored on a hard disk. The float package will be self-contained except for power and supply of liquid helium.

The completed dewar float package will be taken on the KC-135 for one or more flights to observe the motion of the liquid in low gravity. The recorded accelerations will be used as an input to a LMSC-developed CFD code that incorporates the two-fluid model of SFHe. The simulation output will be compared to the actual fluid motion recorded during the KC-135 flight to verify the accuracy of the computer mode.

#### TASK SIGNIFICANCE:

The final product of the project will be to have a CFD code for SFHe that has been verified in low gravity. This will allow predictions of SFHe behavior on future satellites such as SIRTf, GP-B, and AXAF with increased confidence in the accuracy of the simulation.

#### PROGRESS DURING FY 1994:

The objective of this contract is to perform low gravity verification tests of a computational fluid dynamics (CFD) program that incorporates the two fluid model of superfluid helium (SFHe).

#### Accomplishments

The original proposal included the fabrication of a complete new SFHe dewar and float package to perform the verification experiments. This task has been simplified due to the availability of an existing SFHe dewar and float package through JPL. The program plan has been modified as a result of discussions with Peter Mason and Bob Chave at JPL to reuse the dewar and some of the existing support equipment while procuring new hardware to improve the robustness and data-taking capabilities of the system. JPL is upgrading this equipment to have it available as a NASA resource for researchers using SFHe with some refurbishment work being done under this contract. The net result is that it should be possible to conduct the first low-gravity experiments a year earlier than originally proposed.

The technology development related to the use of Lexan as the structural material for the SFHe dewar is proceeding with the objective of providing a replacement unit for the inner SFHe test cell for the existing dewar. A bonded joint between the stainless steel vent lines and the Lexan body has been designed and tested with no leaks after thermally cycling the joint to liquid nitrogen temperatures. The SFHe test cell has been designed and is being fabricated. It will be thermally cycled, proof pressure tested, leak tested, and tested with SFHe as a separate unit; it will then be incorporated into the JPL dewar for laboratory tests and possible use in later low-gravity experiments.

A change has also been made in the computer resources to be used to run the CFD code for this project. This change was induced by accounting changes at Lockheed that now have computer usage costs charged directly to the contract rather than treated as indirect costs. JPL will be making their Cray computer available at no cost to this project. There will be licensing charges from the software vendor for use of the base code on a new machine, but this is anticipated to be relatively low compared to the contract cost of running the code on the Lockheed Cray.

#### Plans

The refurbishment and upgrade of the JPL dewar and float package is planned to be completed by the end of December with the hope of performing the first low-gravity experiments on the DC-9 out of the NASA Lewis Research Center in February.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/93    EXPIRATION: 10/96

PROJECT IDENTIFICATION: 962-24-07-18

RESPONSIBLE CENTER:    JPL

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#### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

##### Proceedings

Ross, G.O. "Helium II slosh in low gravity." Proceedings of the 2nd Microgravity Dynamics Conference.

Ross, G.O. "Behavior of superfluid helium in low gravity." Proceedings of the NASA/JPL1994 Microgravity Low Temperature Physics Workshop.

*Microscale Modeling of Microgravity Multiphase Flow*

PRINCIPAL INVESTIGATOR: Dr. Paul H. Rothe

Creare, Inc.

CO-INVESTIGATORS:

Dr. G.B. Wallis

Dartmouth College

**TASK OBJECTIVE:**

The general objective is to investigate the feasibility of modeling adiabatic multiphase flows on Earth in facilities of very small scale. The specific objective of this contracted effort are to build, instrument, and test a small multiphase flow loop on the order of 1 mm in diameter. This loop -- designed and operated for appropriate flow conditions and geometries -- will potentially scale larger space-based multiphase flow systems.

**TASK DESCRIPTION:**

The principal investigator (PI) will identify, via the appropriate scaling argument, the important non-dimensional quantities governing multiphase flows. The PI will demonstrate, by dynamic similitude, how closely on-orbit conditions can be simulated with this tiny system. A system that is appropriately sized for the proper range of flow conditions will then be designed and built. A comparison will be made between data from this loop and the following: representative data already available from ground tests and low-gravity aircraft tests at larger sizes, existing theory, and in-house numerical models. The relevant data pertain to flow regime and pressure drop predictions.

**TASK SIGNIFICANCE:**

A successful effort will demonstrate a method by which relatively expensive on-orbit, low-gravity, multiphase flow experiments can be complimented, or even simulated, by low-cost, earth-based microscale experiments. Enhanced knowledge will contribute to the design of multiphase thermal control systems being planned for satellite systems, on-orbit space stations, and even moon/planetary applications.

**PROGRESS DURING FY 1994:**

The investigators have completed a feasibility study of using tiny "microscale" facilities on Earth to model multiphase flow in microgravity. Research work was completed early in FY94. The task final report was completed and delivered in March, 1994.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 12/92 EXPIRATION: 6/94

PROJECT IDENTIFICATION: 962-24-05-70

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**NASA Conference Publications**

Rothe, P.H., Martin, C., and Dawning, J. An application of miniscale experiment on Earth to refine microgravity analysis of adiabatic multiphase flow in space. NASA Conference Publication, 3276, (June 1994).

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*Gas Flow from Porous Media and Microgravity Battery Spills*

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PRINCIPAL INVESTIGATOR: Dr. Robert T. Ruggeri

Boeing Company

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

This proposal is based on the hypothesis that spacecraft battery failures can be traced to electrolyte spills resulting from gas expanding inside the capillary pores of battery electrodes. The specific objectives of this project are as follows:

1. To determine the gas capacity of three porous metals as a function of pore volume and pore diameter
2. To determine the system temperature and pressure effect on gas capacity.
3. To determine the effect due to the external geometry of the porous material.
4. Analytically determine the effect of gravity on electrolyte spills.

**TASK DESCRIPTION:**

The objectives will be accomplished by one-g experiments and analysis. The PI will identify all critical parameters that determine a porous metal's gas capacity, then demonstrate, by measurement, the effective volume of noncondensable gas contained within the pore structure of porous metal plates as a function of the identified critical parameters. Such critical parameters are expected to be pore diameter, pore volume, sample shape, temperature, pressure and surface tension. The pore volume and the effective gas capacity of three metals will be determined in volumetric gas flow experiments. The experiments will be conducted at 25°, 45°, and 60 °C in a constant temperature chamber. Zinc, silver, and nickel are the three metals that will be investigated. The effect of pressure cycles will span a range between 14.5 psia and 2.9 psia for each temperature. One to three pressure cycles will be performed for each metal specimen at each temperature. Numerical models will be developed to study the gas capacity as a function of the pore diameter, the temperature, and the ambient pressure. The effect of gravity on these experiments will be determined analytically.

**TASK SIGNIFICANCE:**

Spacecraft batteries have been documented to discharge electrolyte, which causes a short circuit. Electrolyte discharging mechanism is not yet fully understood. In order to prevent mission failures in the future, critical parameters which determine a porous metal's ability to hold and discharge gas should be investigated. This will lead to design of batteries that can retain electrolyte in the microgravity environment of space and thus prevent battery failure in space missions.

**PROGRESS DURING FY 1994:**

During this reporting period, the following have been accomplished:

1. The contract with Boeing Company was officially started on the first of October. A kick-off meeting was held at LeRC on November 8, 1994. Project tasks were reviewed and the approach was discussed.
2. The hardware for the ground-based 1-g experiment were acquired. Setup and checkout will take place in the next few months.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/94    EXPIRATION: 10/96

PROJECT IDENTIFICATION: 962-24-05-95

RESPONSIBLE CENTER: LeRC

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*Ground Based Studies of Thermocapillary Flows in Levitated Drop*

PRINCIPAL INVESTIGATOR: Prof. Satwindar S. Sadhal

University of Southern California

CO-INVESTIGATORS:

E.H. Trinh

Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE:**

For the measurement of the thermophysical properties of undercooled liquids, the idea of spot-heating a test sample in a levitated state is to be explored and applied to relevant materials. That is, a liquid drop levitated in an acoustic field could be heated on a small fraction of its area by a laser beam. In addition, simple filament heating will also be carried out, since this can be achieved with considerable ease. The physical interference of the filament with the fluid mechanics will of course have to be taken into consideration. By carrying out the measurements of the thermocapillary effects of such heating it is possible to derive the thermal properties of the sample. However, this can only be done with the development of a successful predictive model of the system. The effort will therefore consist of both experimental and analytical work.

**1.1 Analytical Part**

The purpose of the analytical part of the proposed program is to develop such a model over several phases. The major thrust at present is in ground based studies with plans for a future space experiment. In the current studies therefore, the experimentation will involve significant interference of the acoustic field. Thus, for most cases for ground based studies, the drops will be deformed to a spheroidal shape. In addition, there is general asymmetry of the flow field. While it is acknowledged that many of these complexities do not arise in low gravity, there is a great deal that can be achieved by ground based studies provided the interference by the acoustic field is fully accounted for in the analysis. For model development in the direction of a zero-g space experiment, analysis will be carried out for liquid shells and compound drops.

Under the scope of the current investigation, the analytical work will consist of several tasks that will encompass the formulation of the differential equations pertaining to levitated drops, their analytical and numerical solutions and the development of results.

**1.2 Experimental Part**

The experimental problem of interest in this proposal is the thermal response of a spot-heated levitated drop in a convective gas flow of varying intensity. The ultimate objective of the tasks proposed is to quantitatively determine the transient and steady-state temperature distribution on the drop surface as a function of time, sample physical properties, geometry, and of the input radiant energy. Because of the coupling of thermocapillary and thermoacoustic phenomena, the interpretation of the resulting thermal state must be carried out in conjunction with the theoretical analysis of the problem. The experimental work will thus be divided into several sub-tasks, each of which must provide data that can be directly correlated with theoretical predictions. Although the final goal will be to carry out an experiment in microgravity, this proposal will limit itself to ground-based investigations using proven experimental techniques in order to correlate with and to verify the theoretical work, as well as to develop experimental methods for a potential future microgravity investigation.

**TASK DESCRIPTION:**

In the usual Earth-based environment, the convective contribution arises due to buoyancy as well as to the effects of the levitation mechanism. In this particular case, the sample may be levitated in a gaseous environment by a high intensity ultrasonic field, and the convective flow field external to the specimen is caused by acoustically-driven streaming flows in addition to the normal buoyancy-driven circulation. The heat transfer problem of determining the transient and steady-state temperature distribution at the surface of the sample will also require the solution of the flow field inside the drop driven by thermocapillary effects (and perhaps also by acoustic radiation stresses) because of the surface tension gradient introduced at the drop surface by the localized heating. Under other

circumstances, the sample may be levitated in a vacuum or gaseous environment by electrostatic forces which do not generate detectable outer convective flows or droplet distortion. This approach requires, however, the permanent non-uniform charging of the drop surface; the effects of which are still unknown, but might also alter the thermally-driven capillary flows.

Under these conditions, the relevant non-dimensional parameters will thus include the Reynolds numbers of the internal thermocapillary-driven flow, of the steady outer acoustic streaming flow, and perhaps of the high frequency acoustic particle motion. The Bond number will be of relevance in order to distinguish between low gravity and Earth-based conditions. The Nusselt, Grashof and Marangoni numbers will also play a primary role. Because we shall be restricted to rather moderate temperature and to conditions far enough from the boiling point of the liquids investigated, mass transfer processes will not be taken into consideration in this case.

#### TASK SIGNIFICANCE:

The proposed research will provide fundamental understanding of the Marangoni flows associated with localized heating of drops and bubbles. For ground based studies where there is interference from the acoustic field, a sound numerical model will provide significant new information about the behavior of these complex systems. The new work on compound drops will play a fundamental role for a zero-g space experiment. Most importantly, the model development along with the experimental studies will represent fundamental groundwork for the measurement of thermophysical properties of undercooled liquids.

#### PROGRESS DURING FY 1994:

##### 1. Experimental Work.

##### 1.1. Outer acoustic streaming flows characteristic of ultrasonic levitators in 1 G:

The ultrasonic streaming flows have been investigated using smoke particle visualization inside empty levitation chambers as well as around levitated isothermal and spot-heated solid and liquid samples. These studies clearly indicate that the convective flow environment around a spot-heated sample is dominated by the interaction between acoustic streaming and natural convection. Different regimes can be identified and vary from laminar, low frequency oscillatory, chaotic, and finally turbulent as the streaming Reynolds number increases. Some of the findings have been summarized in a publication by Trinh and Robey (1994).

##### 1.2. Spot heating of a levitated drop in the ultrasonic-electrostatic hybrid levitator:

A pulsed CO<sub>2</sub> laser has been used to spot heat levitated charged drops with radii on the order of 4-5 mm in diameter. Heating from one side has been observed to generate the steady rotation of an initially stationary spherical drop with angular velocity vector in the vertical direction. The thermal distribution at the drop surface has been monitored with an Infrared imaging camera with a 0.1 °C resolution. The elimination of rotation by imparting an opposed and controlled acoustic torque is being implemented together with an apparatus allowing heating along the symmetry axis in the vertical direction.

##### 1.3. The visualization of internal flows with and without radiant heating:

Tracer particle scattering and particle image velocimetry (PIV) methods are being implemented to obtain quantitative internal flow velocity for the axisymmetric and steady case. A background internal flow field for electrostatically levitated charged drops in the presence of a weak ultrasonic field has been measured, and a typical velocity is on the order of 0.05 cm/sec.

##### 2. Analytical Work:

##### 2.1 Thermocapillary Flows in a Spot-Heated Oblate Spheroid

From the previous pure conduction calculations in the case of axisymmetric spot heating of an oblate spheroid, the

thermal driving force at the interface has been applied to the calculation of the flow field. This has led to an elaborate expression for the stream function and numerical calculations are being carried out. Since these results are limited to the steady state and a weak thermal driving force, further progress will require fully numerical schemes which will be developed.

## 2.2 Outer Acoustic Streaming Around a Spheroid

Following an earlier analysis of Lee and Wang on the problem of streaming around a sphere, the generalization to an oblate spheroid has been carried out. The principal result is the development of the tangential component of the fluid velocity outside the boundary layer. This has led to the complete solution of the Stokes flow field around the inner boundary layer region. Numerical calculations have yet to be carried out.

## 2.3 Thermoacoustic Streaming from a Sphere with Pulsating Spot Heating

Following the singular perturbation scheme of Gopinath and Sadhal (1994), for small values of the streaming Reynolds number, an extension to the case of pulsating spot heating has been developed. The frequency of oscillation is in tune with the acoustic field. Physically, the situation is achievable by means of a laser beam directed towards a spherical sample. The thermal energy deposited is divided between the solid sphere and the surrounding gas medium on the basis of the respective thermal transport characteristics. With a pulsating heat source resonating with the acoustic frequency, the time averaged energy equation yields a nonzero convective transport. Some analytical difficulties have prevented the completion of this work. These will be resolved within the next few months.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-07-19

RESPONSIBLE CENTER: JPL

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Trinh, E.H. and J.L. Robey. Experimental study of streaming flows associated with ultrasonic levitators. Phys. of Fluids. (To be published November 1994).

Trinh, E.H. and K. Ohsaka. Measurement of density, sound velocity, surface tension, and viscosity of freely suspended supercooled liquids. International Journal of Thermophysics, (accepted for publication).

#### Proceedings

Gopinath, A. and S.S. Sadhal. "Thermoacoustic streaming effects from a sphere subject to a time-periodic temperature disturbance." Heat Transfer 1994, Vol. 5, pages 279-284. Tenth International Heat Transfer Conference, Brighton, UK, August 1994.

#### Books

"Heat Transfer in Microgravity Systems - 1993." Edited by: S.S. Sadhal and A. Hashemi American Society of Mechanical Engineers, 29th National Heat Transfer Conference, Atlanta, Vol. HTD-235, August, 1993.

"Heat Transfer in Microgravity Systems II - 1994." Edited by: S.S. Sadhal and A. Gopinath American Society of Mechanical Engineers, International Mechanical Engineering Congress and Exhibition, Chicago, IL, Vol. HTD-295, November 1994.

#### Presentations

Trinh, E.H. and S.S. Sadhal. "Acoustic streaming and ultrasonic processing of low melting point materials." 1994 ASME International Mechanical Engineering Congress and Exhibition, November 1994.

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*Effects of Gravity and Shear on the Dynamics and Stability of Particulate and Multiphase Flows*

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PRINCIPAL INVESTIGATOR: Prof. Ashok S. Sangani

Syracuse University

## CO-INVESTIGATORS:

Donald L. Koch

Cornell University

Michel Louge

Cornell University

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TASK OBJECTIVE:

To understand the particulate and multiphase flow behaviors and dynamics that will occur in the microgravity and Earth's gravity environments, and to investigate systems in which the inertial effects are important on the length scale of particles and bubbles.

## TASK DESCRIPTION:

Complementary theoretical, simulation and experimental approaches to achieve the aforementioned task objective. The study will consider two types of inertial suspensions that are amenable to detailed theoretical studies. The first type is a solid-gas suspension, in which the inertia of the particle and the gas viscosity are more important than the gas phase inertia. The second type is a suspension of bubbles with high Reynolds number indicating inviscid flow, but with small Weber number indicating that their deformation is small. Theoretical development of sheared particulate and bubbly liquids will make use of concepts borrowed from the kinetic theory which has been successfully applied to granular flows. The kinetic theory will be complemented by a numerical simulation of the solid-gas and gas-liquid suspensions. These simulations include detailed calculation of the hydrodynamic interactions among the particles in both microgravity and Earth gravity conditions. To validate the equation of motion of the suspension systems, experimental measurements of suspension properties in simple basic flow situations are necessary. The initial earth-based experiment will involve sheared fluidized bed by using capacitance probes to measure the volume fraction as a function of time, the shear rate required to overcome the gravitational instability. The knowledge gained from the theoretical, simulation, and experimental work will set the necessary background to propose low gravity experiments.

## TASK SIGNIFICANCE:

Shearing fluidized bed and bubbly suspensions are used in chemical processing and understanding the dynamics and flow behavior in these systems is crucial.

## PROGRESS DURING FY 1994:

The aim of this project is to study, both experimentally and analytically, the flows of two suspensions with significant inertial effects: the gas-solid suspensions with vanishingly small Reynolds number and finite Stokes number,  $St$ , and bubbly liquids at large Reynolds number. During the first three months of the project (06/24/94-10/04/94), we have completed an analytic investigation of the simple shear flow of dilute gas-solid suspension. We found that the suspension exhibits multiple steady states for range of values of  $St$  and the volume fraction  $f$ . The final steady state depends on the initial conditions of the suspension. For sufficiently large initial velocities, the final state, which we refer to as the ignited state, has a very large velocity variance, and for small initial variance the final state is quenched with a vanishingly small final variance. A kinetic theory has been developed to explain these and several other interesting results observed in the numerical simulations of the flow. We are currently extending this theory to larger  $f$  and comparing it to the results of Stokesian dynamic simulations of these suspensions.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/94    EXPIRATION: 5/96

PROJECT IDENTIFICATION: 962-24-05-96

RESPONSIBLE CENTER: LeRC

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*Dielectric and Electrohydrodynamic Properties of Suspensions*

PRINCIPAL INVESTIGATOR: Dr. Dudley A. Saville

Princeton University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

This investigation focuses on understanding those electrokinetic properties of particulate suspensions related to the so-called "electrohydrodynamic effect", specifically, the dielectric constant and electrical conductivity. These properties are of crucial importance in defining the behavior of samples in various electrokinetic separation processes. The work involves two research tasks:

1. Measurements of the electrokinetic properties of a series of suspensions with particle volume fractions between 1% and 20% by volume.
2. Development of a theory for the dielectric constant and conductivity of suspensions which encompasses the measured behavior.

**TASK DESCRIPTION:**

**Experimental work:** We have been attempting to prepare model particles which conform to the classical theory. Previous research showed that with some particles annealing at 120 °C (above the glass transition temperature) smoothed the particle surface so suspensions behaved according to the classical theory. Attempts to prepare such particles using both anionic and amphoteric latexes purchased from the Interfacial Dynamics Corporation, met with limited success. The reasons for this behavior are not at all clear. Accordingly, we have been working to adsorb polymer on latex particles to increase their dipole moment. Suspensions prepared with these particles will have large dielectric constants which can be controlled by the amount and size of the adsorbed polymer.

**Theoretical work:** We continue to work on theoretical models of the electrohydrodynamic effect and on models of the electrokinetic behavior of dispersions. The purpose of these efforts is to provide the requisite theory to interpret our results.

**TASK SIGNIFICANCE:**

This research is intended to develop an understanding of one of the major obstacles to effective separation of particles by electrokinetic methods. During the past year we uncovered ways of adapting our techniques to understanding the behavior of particles in non-aqueous systems. Thus, in addition to its use in separations, our work should find applications in ceramic processing.

**PROGRESS DURING FY 1994:**

During the past year significant progress was made as follows:

1. We successfully measured the dielectric properties of suspensions containing particles with adsorbed polymer (polyethylene oxide). As expected these suspensions had significantly higher dielectric constants. They may provide an excellent model system with which to study electrohydrodynamic effects.
2. The dielectric properties of concentrated dispersions were measured at volume fractions up to 30 %.
3. A theory for the macroscopic hydrodynamic behavior was developed (and published).

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 6/91 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-24-08-08

NASA CONTRACT NO.: NAG8-878

RESPONSIBLE CENTER: MSFC

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Saville, D.A., Rosen, L.A., and Baygents, J.C. Dielectric relaxation in dispersions of colloidal particles with a dynamic Stern layer. *J. Chemical Physics*, vol. 98, 4183-4194 (1993).

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Saville, D.A. "Electrohydrodynamic deformation of a particulate sample." *AICHE Annual Meeting*, St. Louis, MO, November, 1993.

Saville, D.A. "The roles of dynamic processes behind the shear plane." *International Colloid and Surface Science Symposium*, Adelaide, South Australia, February, 1994.

Saville, D.A. "Electrokinetic behavior of concentrated colloidal dispersions." *ACS Colloid Symposium*, Stanford, CA, June, 1994.

Saville, D.A. "Dielectric relaxation in suspensions of particles with physisorbed PEO." *ACS Colloid Symposium*, Stanford, CA, June, 1994.

*Electrohydrodynamic Pool Boiling in Reduced Gravity*

PRINCIPAL INVESTIGATOR: Prof. Benjamin D. Shaw

University of California, Davis

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

To investigate the effects of electric fields on reduced-gravity pool boiling. The electric fields are expected to significantly and controllably increase reduced-gravity nucleate boiling rates and maximum heat fluxes. The presence of an electric field will result in the production of smaller bubbles in reduced gravity and the average speed will be increased.

**TASK DESCRIPTION:**

A drop apparatus will be constructed for use at the NASA Lewis 2.2 Second Drop Tower. This apparatus will consist of a pool boiling test chamber and associated instrumentation mounted on a NASA drop frame. Boiling will occur on an electrically-heated platinum wire subjected to a nonuniform external DC electric field. Boiling experiments will R-113 (trichlorotrifluoroethane,  $(CCl_2FCIF_2)$ , for which  $t_c \gg t_b$ ) or water (for which  $t_c \ll t_b$ ) will be performed in both 1-g and  $\mu g$ . Data will be gathered on applied electric fields and wire heat fluxes and temperatures. High-speed motion picture photography will provide visual records of boiling phenomena.

**TASK SIGNIFICANCE:**

Anyone who has ever boiled water on a stove is familiar with nucleate pool boiling. Even though it is an everyday event, scientists do not understand precisely how it works, because the Earth's gravity influences how bubbles form and grow in boiling liquids.

NASA is interested in the results from this experiment, because boiling liquids generate bubbles which are very efficient at transferring large amounts of heat. Finding new ways to dissipate heat from the space shuttle or future manned space platforms will be vital to the success of long-term missions.

The potential benefits closer to home, including more effective air conditioning and refrigeration systems, and improvements in power plants that could reduce the cost of generating electricity.

**PROGRESS DURING FY 1994:**

Funding for this cooperative agreement began mid June 1994. Future plans include the following:

July 1994 to July 1995: Construct pool boiling apparatus and conduct 1g tests.

July 1995 to July 1996: Conduct reduced gravity tests in LeRC 2.2 Second Drop Tower in two 5-week sessions, (50 to 100 drops are planned) and analyze and report results.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 6/94 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-24-05-97

RESPONSIBLE CENTER: LeRC



*Transport Processes Research*

PRINCIPAL INVESTIGATOR: Dr. Bhim S. Singh

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this task is to promote, foster, and enhance the quality and breadth of microgravity research conducted in the discipline of fluid physics by advancing the understanding of thermal and mass transport processes when buoyancy-driven convection is reduced or eliminated.

**TASK DESCRIPTION:**

The approach to achieving the task objective is to provide LeRC in-house support to assist sponsored principal investigators in the conduct of their research (particularly when that research can benefit from unique expertise or facilities at LeRC), while guiding and assisting in the definition of flight experiments.

**TASK SIGNIFICANCE:**

This task will assist in program planning and outreach programs in the external community, and it will conduct in-house research to advance the understanding of transport and interfacial phenomena through exploitation of the microgravity environment.

**PROGRESS DURING FY 1994:**

A linear stability analysis on Marangoni-Benard convection in a liquid layer bounded by a free surface from below and a free surface from above has been conducted for both non-deformable-free-surface case and deformable-free-surface case. The results show that the instability is surface wave instability in nature, rather than hydrodynamic instability. It is also shown that surface deformation changes the characteristics of the Marangoni-Benard instability to long wavelength unstable. Oscillatory instability occurs only at near the long wavelength end ( $k \ll 1$ ), and that higher Prandtl number tends to reduce the domain of oscillatory instability. On the experiment side, the bottom heater was upgraded by implementing two high-resolution thermistors, one at the center and one at near the edge. A precision thermal controller was also purchased and used. Initial experiments on the Marangoni Instability in a liquid sheet with 2 free surfaces are being conducted with a silicone oil layer supported by a layer of FC75 from below and exposed to an air layer on top.

The coupled Marangoni-Benard/Rayleigh-Benard problem with temperature dependent viscosity was solved as a numerical eigenvalue problem. In this study, the reference temperature is taken to be temperature of the bottom surface and a linear (first order approximation) viscosity profile with respect to temperature is used. The temperature dependent viscosity was found to be stabilizing for the Rayleigh-Benard Problem, the Marangoni-Benard Problem, and the coupled problem. The linear viscosity profile was shown to be valid for typical Benard studies of thin liquid layers. The choice of reference temperature influence the variable viscosity effect. Results are currently being summarized.

The performance of a spectral (Chebyshev collocation) formulation of the benard problem has been compared with a finite (central) difference formulation of the same problem. Results of both discrete formulations are compared to exact solutions of the Benard problem. The error of the spectral scheme is at least seven orders of magnitude smaller than the finite difference error for a grid resolution of  $N=15$  (number of points used).

In the area of thermocapillary bubble/drop migration, higher order asymptotic results for the bubble velocity have been rederived following a slightly modified approach in the analysis of the outer temperature field. This was done

in response to comments from referees. Analysis was continued to determine the temperature field inside a migrating drop when convective effects are predominant. Numerical efforts to model the same problem are also under way.

Tests to examine capillary driven flows in wedges and corners were continued using the drop tower. An analytical model for capillary flow in corners was developed.

Measurement capabilities of the LeRC fluid physics laboratories were used to support multiple flight definition PI's. Measurements of thermophysical properties e.g. surface tension of Newtonian and non-Newtonian fluids were performed. Optical techniques were developed/improved to provide better flow visualization and quantitative measurements. These include two- and three-dimensional particle image velocimetry and point diffraction interferometry.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 2  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 1/94 EXPIRATION: 1/95

PROJECT IDENTIFICATION: 962-24-05-01

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Balasubramaniam, R. Thermocapillary bubble migration: solution of the energy equation for potential-flow approximated velocity fields. Japan Society for Computational Fluid Dynamics (in press), (1994).

Duh, J.C. Marangoni-Bernard convection in a low-aspect-ratio liquid layer. J. Microgravity Science and Technology, vol. 7, 98-102 (1994).

#### Proceedings

Skarda, J.R.L., McCaughan, F.E. and Fitzmaurice, N. "The Bernard Problem: A comparison of finite difference and spectral collocation eigenvalue solutions." 6th Annual Thermal and Fluids Analysis Workshop, NASA LeRC/NASA CP-10161, August 1994.

#### Presentations

Duh, J.C., Chien, L. and To, K. "Post-onset evolution of marangoni-bernard convection in a liquid layer heated from below." 45th International Astronautical Federation Congress, Jerusalem, Israel, October 9-14, 1994.

Oron, A., Deissler, R. and Duh, J.C. "Marangoni instabilities in a liquid sheet." 30th COSPAR Scientific Assembly, Hamburg, Germany, July 11-17, 1994.

Skarda, J.R.L. and McCaughan, F.E. "Coupled Marangoni-Bernard/Rayleigh-Bernard instability with temperature dependent viscosity." ASME Winter annual meeting, Chicago, IL/NASA TM 106646, November 1994.

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*Solute Nucleation and Growth in Supercritical Fluid Mixtures*

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PRINCIPAL INVESTIGATOR: Dr. Gregory T. Smedley

California Institute of Technology

CO-INVESTIGATORS:

Gerald Wilemski

Lawrence Livermore National Laboratory

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**TASK OBJECTIVE:**

We plan to study the behavior of naphthalene in supercritical carbon dioxide due to the availability of thermodynamic data for this system, the accessibility of relevant experimental conditions in the laboratory, and the importance of CO<sub>2</sub> in many technological applications. The strong dependence of solubility on pressure enables the nucleation and growth processes to be experimentally decoupled, so they may be studied independently.

**TASK DESCRIPTION:**

1. Determine experimental parameters for supercritical expansion processes. These include thermodynamic process paths, equilibrium solubilities, particle size and growth rate estimates.
2. Make optical measurements of solid particle nucleation and growth rates in fluid mixtures of CO<sub>2</sub> and naphthalene that are initially prepared in a supercritical state.
3. Analyze experimental data, including phenomenological analytical modeling as necessary.
4. Develop and recommend a flight experiment design based on the results of the ground-based experiment.

**TASK SIGNIFICANCE:**

The experiments will employ rapid, but controlled, changes of the supercritical fluid pressure to vary the thermodynamic state of the fluid from a stable condition to one favoring solute nucleation and then to one fostering only solute particle growth. By using optical methods to measure the number of particles formed and their size, we can determine nucleation rates and growth rates at various thermodynamic conditions. Since the rates of nucleation of solid particles have never before been measured under these conditions, the data to be obtained will be invaluable for testing and improving nucleation theory as well as for guiding the design of a space-based experiment. One of the most important goals of the ground-based experiment will be to evaluate the effects of gravity on the measured nucleation rates. Because the density of near- and supercritical fluids varies rapidly with pressure, gravity may induce nonuniform conditions in the experimental fluid mixtures. This would make accurate ground-based measurements difficult if not impossible.

The results of this experimental/analytical work can be applied to the understanding of nucleation and growth problems found in many fields such as biotechnology (protein crystal growth), material science (sintered alloys), and analytical chemistry (supercritical fluid extraction and chromatography).

**PROGRESS DURING FY 1994:**

The necessary contractual arrangements have been processed to fund this proposed research which was accepted under the 1991 NRA. Funding began in August, 1994. The research effort has just been initiated.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 8/94 EXPIRATION: 8/97

PROJECT IDENTIFICATION: 962-24-05-69

RESPONSIBLE CENTER: LeRC

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### *Behavior of Unsteady Thermocapillary Flows*

PRINCIPAL INVESTIGATOR: Prof. Marc K. Smith

Georgia Institute of Technology

CO-INVESTIGATORS:

Prof. J.N. Koster

University of Colorado

#### TASK OBJECTIVE:

The objective is to investigate thermocapillary instabilities to provide an understanding of the mechanisms of flow instabilities. The study will also indicate how well the stability theory results for simple geometries apply to the behavior of thermocapillary flows in more complex geometries.

#### TASK DESCRIPTION:

The research approach consists of both a theoretical and an experimental effort. Two nonlinear analytical models for study of post-critical thermocapillary flows in a bounded domain will be constructed as part of the theoretical work. The models will be used to explore the effects of  $Pr$ , interfacial heat transfer, domain size, 3-D disturbances, and interfacial deformation on system flow stability. An experimental investigation of thermocapillary instabilities of opaque, low  $Pr$  and high  $Pr$  fluids will be also be performed.

#### TASK SIGNIFICANCE:

This understanding will assist fluid system designers optimize system designs in which thermocapillary flows are important.

#### PROGRESS DURING FY 1994:

##### Theoretical Work:

In the second year of this project, we have continued our analysis of the behavior of the thermocapillary flow of a liquid layer contained in a shallow cavity. Previously, we have used asymptotic techniques appropriate to very thin layers to derive a nonlinear partial differential equation describing the evolution of the free surface of the liquid layer. We have solved this evolution equation numerically for a two-dimensional steady flow. The solution is parameterized in terms of a modified capillary number that measures the effect of surface tension. For small capillary numbers (large surface tension), the free surface of the layer is almost flat. As the capillary number increases, the deformation of the interface increases with a bulge forming near the cold end of the cavity and a depression occurring near the hot end. This behavior is in keeping with asymptotic solutions found previously by Sen and Davis (1982).

This year our numerical calculations have indicated that when the modified capillary number exceeds 43.3, no steady solution exists. We have used an arc length continuation technique (the AUTO94 software package) to trace the solution curve past this limit point. The curve doubles back on itself and indicates that for capillary numbers less than the critical value of 43.3, there are actually two distinct solutions to the steady flow in the cavity. The solution on the upper branch has much more interfacial deformation than the lower branch. For the smaller values of capillary number, the deformation reaches the total depth of the layer and so we must stop the calculations because the layer most probably ruptures. Our evolution equation does not apply in this situation.

We have also done a stability analysis of the steady solutions on this entire solution curve. The solutions on the lower branch, in which the deformation is smaller, are linearly stable. The solutions on the upper branch are linearly unstable.

Our current direction is to do a time dependent calculation of the behavior of the layer. One can easily speculate that for the unstable upper branch, the layer will move toward the stable branch solution for the same capillary number

when the layer is disturbed. However, for small values of the capillary number it is possible that the layer could increase its deformation and approach a ruptured state in which liquid is contained in each corner of the cavity and a dry spot appears in between. For larger values of the capillary number where steady solutions do not even exist, an initially uniform liquid layer will most probably deform continuously until it either ruptures in the middle or it comes to a new state in which the thin-film physics forces the layer to remain continuous. The appropriate physics required for this behavior has yet to be included in our model of the liquid layer.

#### Experimental Work:

The experimental work has been conducted at the University of Colorado at Boulder under the direction of Prof. Jean N. Koster. The experiments using optical techniques on silicone oils have been completed. The optical test cell is an open, rectangular container in which the liquid layer measures 92 mm long by 15 mm wide. The two ends of the cell are copper and are held at different fixed temperatures. The lower surface is PVC which simulates an insulated surface and the side walls are made of glass. The container was filled with a layer of silicone oil starting at a depth of 1 mm and increasing up to 4 mm in increments of 0.5 mm. This oil was driven in a thermocapillary motion by the temperature difference of the ends of the cell. Two different oils were used; their Prandtl numbers were 8.5 and 27.5. The free-surface deflection of the layer was observed by means of strioscopy.

The results of the experiments showed that an instability occur for a temperature gradient across the layer above a critical value. For small layer depths, the instability takes the form of a regular train of waves propagating from the hot end to the cold end. These may be an example of a hydrothermal wave predicted by Smith and Davis (1983a). For larger depths, an irregular train of waves appears that also propagates in the same direction. The critical Marangoni numbers for these transitions are higher than the theoretical values computed by Smith and Davis (1983a). The discrepancy is probably due to the neglect of gravity in the theory and the different geometry of the experiments.

The flow patterns in the layer were visualized by seeding the layer with particles and taking streakline photographs of the flow. For a thin layer and with the imposed temperature gradient just less than the critical value, the flow field takes the form of a multi-cellular steady state. When the temperature gradient is greater than the critical value and the regular wave train appears, there seems to be no change in the multi-cellular flow field inside the layer. For a thicker layer and with the imposed temperature gradient just less than the critical value, the flow field takes the form of a single, steady recirculation cell and a much smaller cell near the hot end. When the temperature gradient is greater than the critical value the irregular wave train appears and a transient behavior of the recirculation cell is observed.

An attempt to use holographic imaging to visualize the flow instability in the layer was made. The results were not very successful because large temperature gradients and the convective flow in the layer would produce significant distortions in the hologram. In the low-viscosity silicone oil layer, the temperature gradients and convective motions were reduced and so some success was achieved. The advantage of using holography is the much higher resolution for the visualization of small free-surface deformations. This increased resolution detected a second instability in the layer in the form of an intermittent wave with a wave speed about twenty times larger than the previous wave-like instability. It may be an example of a surface wave predicted by Smith and Davis (1983b).

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-24-05-62

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

Sen, A.K. and Davis, S.H. Steady thermocapillary flows in two-dimensional slots. J. Fluid Mech. 121, 163-186 (1994).

Smith, M.K. and Davis, S.H. Instabilities of dynamic thermocapillary liquid layers, part 1, convective instabilities. J. Fluid Mech. 132, 119-162 (1994).

Smith, M.K. and Davis, S.H. Instabilities of dynamic thermocapillary liquid layers, part 2, surface-wave instabilities. J. Fluid Mech. 132, 145-162 (1994).

*Flow-Influenced Shape Stability: Breakup in Low Gravity*

PRINCIPAL INVESTIGATOR: Prof. Paul H. Steen

Cornell University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

1. Understand the influence in low gravity of flow on interface shape. For example, document and control the influence of axial flow on the Plateau-Rayleigh instability of a liquid bridge.
2. Extend the ground-based density-matching technique of low gravity simulation to situations with flow; i.e., develop Plateau chamber experiments for which flow can be controlled.

**TASK DESCRIPTION:**

The configuration of a liquid held by surface tension under low gravity is susceptible to significant modification by liquid motion. Motion destabilizes in general but there are narrow circumstances where motion can stabilize. We identify these circumstances theoretically, by solving linear and nonlinear stability problems, and try to locate them in experiments conducted with a dynamic Plateau apparatus. Overall, the goal is an understanding of the influence of flow on shape.

**TASK SIGNIFICANCE:**

Containerless containment of liquids by surface tension has broad importance in low gravity. For space vehicles, the behavior of liquid/gas interfaces is crucial to successful liquid management systems. In microgravity science, free interfaces are exploited in various applications. Examples include float-zone crystal growth, phase separation near the critical point of liquid mixtures (spinodal decomposition) and quenching of miscibility gap molten metal alloys. In some cases, it is desired to stabilize the capillary instability while in others it is desired to induce capillary breakup. In all cases, understanding the stability of interface shape in the presence of liquid motion is central.

**PROGRESS DURING FY 1994:**

Both analytical/numerical and experimental approaches are employed.

Stability analyses include linear and nonlinear techniques. The linear stability approach has been used to analyze the shape stability of a cylindrical interface containing axial shear flows, both isothermally and thermocapillary-driven. Computational feasibility currently limits this approach to base states that are separable flows, effectively, the axial-infinite interfaces. It is now well-known that infinite cylindrical interfaces can be stabilized. For finite interfaces an alternative approach is needed. In the limit of no motion, minima of the free-energy functional are obtained using the calculus of variations supplemented by numerical branch-tracing. For weak motion (creeping flow), we extended this approach below using a modified functional. Near the singularity represented by the Plateau-Rayleigh limit, bifurcation theory using Liapunov-Schmidt reduction is a natural tool for the solution of the appropriate nonlinear Euler-Lagrange equation. All these analytical/numerical tools lend themselves to understanding the physics of stability in terms of simple competition mechanisms.

As for the experimental approach, a dynamic Plateau chamber has been built and is used to study liquid bridges held captive by rod-ends and embedded in a controlled surrounding flow. Theory has guided the experiments to a particular window in parameter space. Such guidance is crucial since interesting stabilization effects occur over narrow parameter ranges for this problem.

### Theory results:

- 1) Stability (instability) of a static bridge equilibrium ( $B = 0$ ) is immediate once the family of equilibria to which it belongs is identified; direct calculation of the second variation is circumvented.
- 2) All known families of static bridge equilibria ( $B = 0$ ) are ultimately connected and thereby inherit their states of instability (number of unstable modes) ultimately from the stability of the sphere.

### Experiment results:

- 3) A comparison of pairs of relatively immiscible liquids suitable for use in a dynamic Plateau chamber with density balance within  $10^{-4}$  g/cm<sup>3</sup> has been presented. Pure water and the isomeric system of 2-, 3- and 4-fluorotoluene is one preferred pair. Pure water and the homologous system of chlorocyclohexane and chlorocyclopentene is another.
- 4) Further observation of analysis of the collapse of the soap-film bridge have been performed. The soap-film collapse is viewed as a prototype collapse.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	1	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 1/93    EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-24-05-57

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Lowry, B.J. and Steen, P.H. Stabilization of an axisymmetric liquid bridge by viscous flow. *Int. J. Multiphase Flow*, 20 (2), (1994).

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Lowry, B.J. and Steen, P.H. On the density matching of liquid using plateau's method. *AIAA paper*, 94-0832, (1994).

#### Proceedings

Chen, Y.J. and Steen, P.H. "Influence of flow on interface shape stability." *Proceedings of the 24th IMACS World Congress*, Atlanta, GA, July 1994.

#### Presentations

Lowry, B.J. "Ph.D. dissertation." Cornell University, Ithaca, NY, 1994.

Lowry, B.J. and Steen, P.H. "Capillary surfaces: stability from families of equilibria with application to the liquid bridge." *Proc. Roy. Soc. A*, accepted.



*Interactions of Bubbles and Drops in a Temperature Gradient*

PRINCIPAL INVESTIGATOR: Prof. R. S. Subramanian

Clarkson University

## CO-INVESTIGATORS:

Dr. R. Balasubramaniam

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objective of this research is to study the interactions of bubbles and drops in the presence of a temperature gradient in the continuous phase. The goal is to understand how the presence of a neighboring drop alters the motion of a test drop and whether the two drops coalesce. Both theoretical and experimental research is planned on the interactions of drops with each other or with a neighboring boundary.

**TASK DESCRIPTION:**

The emphasis of the initial effort is on the design, fabrication and set up of the experimental hardware, and to check its performance. Experiments will then be performed using a suitably chosen liquid-liquid system such that the drops sink due to buoyancy. The drops will be subjected to a vertical temperature gradient; thermocapillary forces will then cause the drops to move upwards. Initially experiments will be performed with single drops and the results will be compared with existing theory. Subsequent experiments will focus on drop interactions.

**TASK SIGNIFICANCE:**

This research attempts to provide important information on interactions of drops, i.e., how the presence of a drop affects the motion of another, in a medium where the temperature is non-uniform. Primary emphasis is on thermocapillary flow, thereby the research is more applicable to drop interactions under reduced gravity. The nature of these pairwise interactions determines whether the drops speed up or slow down as they approach each other and whether they collide or glide past each other. Pairwise interaction data are very useful in models that track coalescence in a many drop dispersion.

**PROGRESS DURING FY 1994:**

Experiments have been conducted on the motion of single drops of diethyl maleate in 1,3 - propanedial. The drop sizes ranged from 0.04 to 0.65 mm and the upward temperature gradients from 0.045 - 2.4 K/mm. The results from these tests clearly demonstrate thermocapillary effects - the drops sink due to buoyancy when the gradient is low and rise when the gradient is high. Dissolution of some species from the drop fluid into the outer fluid has been observed and steps have been taken to minimize its impact. For situations where the convective transport of energy is negligible, the data is in agreement with theoretical predictions. New theory is needed to compare with the experimental results with significant convective energy transport effects.

A new test cell has been designed and constructed. The design accommodates the insertion of pairs of drops and viewing their motion from two orthogonal directions. Preparatory tests to check the performance of the test cell are under way and interaction studies are expected to commence soon.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 3/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-24-05-58

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Presentations**

Lacy, C.E., Subramanian, R.S., Balasubramanian, R. "The behavior of drops in a vertical temperature gradient - influence of convective transport of energy." to be presented at the AIChE 87th Annual Meeting, San Francisco, November 1994.

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*Instability in Surface-Tension-Driven Benard Convection*

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PRINCIPAL INVESTIGATOR: Prof. Harry L. Swinney

University of Texas, Austin

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## CO-INVESTIGATORS:

Prof. W.D. McCormick

University of Texas, Austin

Prof. J.B. Swift

University of Texas, Austin

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**TASK OBJECTIVE:**

The objectives of this work are to investigate the primary and secondary instabilities in surface-tension driven convection and in double-diffusive convection in the Hele-Shaw geometry, and to characterize both the global structures and the local flow properties of Benard Convection.

**TASK DESCRIPTION:**

Noninvasive optical techniques will be developed and employed to study the primary and secondary instabilities over a wide range of Marangoni numbers. Theoretically, nonlinear analyses for the long wave ( $k=0$ ) instability will be conducted with surface deflection included. Direct numerical simulation of the two- and three-dimensional incompressible fluid equations will be used to investigate both weakly non-linear behavior near the primary instability and secondary instabilities in Marangoni convection.

**TASK SIGNIFICANCE:**

Surface tension force dominates many physical processes in space and strongly influences many important practical problems on Earth, such as the processing of commercially important materials (electronic or biological crystal growth), manufacturing (welding, coatings), as well as industrial heating, cooling, and mixing (thin film heat exchangers). This work seeks to elucidate clearly the basic mechanisms that can cause fluids to change from a simple quiescent state to a turbulent behavior. Such a transition can have a dramatic effect on the practical problems described above. Furthermore, in the course of this work, new experimental techniques such as the enhanced infrared imager will be developed that may later find wider use in other space/terrestrial applications of commercial importance.

**PROGRESS DURING FY 1994:**

In FY94, experimental evidence was obtained for the existence of hysteresis at the onset of hexagonal convection. These results suggest that the primary instability in surface-tension-driven Benard convection can occur as a subcritical bifurcation, as predicted by theory. Surface temperature distribution in Benard convection is being measured using a state-of-the-art infrared imager. Techniques were developed to optimize the spatial and temperature sensitivity of the measurements. A new apparatus was designed and built to permit infrared imaging of the flow field while maintaining good temperature control in the convection cell. In addition, the long wavelength instability in Benard convection, which may dominate in microgravity, was investigated theoretically. A nonlinear evolution equation in two-dimensions was derived; moreover, an analysis of this equation demonstrates the primary bifurcation to the long wavelength mode occurs subcritically for all parameters investigated. Use of a purified single component silicone oil was demonstrated to eliminate deleterious evaporation/condensation effects in the experiment. A protocol for systematically purifying commercial silicone oils for use in convection experiments is being investigated.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 4  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-05-41

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### **Presentations**

Schatz, M. "Onset of hexagons in surface tension driven Benard convection." 2nd Microgravity Fluid Physics Conference, NASA Lewis research Center, June, 1994.

Schatz, M. "Benard convection: New experimental techniques." Texas Section of the American Physical Society, October, 1994.

Schatz, M. "Infrared Imaging of temperature in Benard convection." Annual Meeting of APS Fluid Dynamics Section, Atlanta, GA, November, 1994.

VanHook, S. "Long Wavelength thermocapillary instability in Benard convection." Annual Meeting of the APS Fluid Dynamics Section, Atlanta, GA, November, 1994.

*Crystal Growth and Fluid Mechanics Problems in Directional Solidification*

PRINCIPAL INVESTIGATOR: Prof. Saleh Tanveer

Ohio State University

## CO-INVESTIGATORS:

Dr. G. R. Baker

Ohio State University

Dr. M. R. Foster

Ohio State University

## TASK OBJECTIVE:

The primary objective of this task will involve efforts to build a comprehensive theory for dendritic crystal growth, with and without fluid convection. This is a difficult task as it involves a nonlinear time-dependent evolution equation that is nearly ill-posed when capillarity effects are small. Bridgman Crystal Growth situations will also be studied.

## TASK DESCRIPTION:

The effort will primarily consist of a numerical study of possible bifurcation of steady-state solutions that may be steady or oscillatory in two or three dimensions.

## TASK SIGNIFICANCE:

It is expected that some interesting subclasses of problems will be found for extreme values of parameters; in these cases, it is possible that the essential phenomena can be captured by a nonlinear analysis of equations that contain only a few parameters.

## PROGRESS DURING FY 1994:

During the funding period of support, Jan 1994 to present, continued progress has been made in two different crystal growth problems - dendritic crystal growth and the Bridgman problem.

## A. Dendritic Crystal Growth

In the small Peclet number asymptotic limit, we have carried out both numerical and asymptotic analysis with a view to understanding some controversial issues in selection theory and in the time development of dendrites.

If we recall, in studying certain classes of disturbances superposed on an initially parabolic dendrite, in the asymptotic limit of small Peclet number  $Pe$ , it was recognized that there are different regions where the equations for the temperature field and boundary conditions have different forms. In an  $O(1)$  region around the dendrite tip, the diffusive field reduces to a Laplacian. With a novel numerical scheme, we have computed highly accurate solutions in the tip region showing the evolution of an initially parabolic dendrite with nonzero surface tension. For a long time, perturbation from the initial parabola is found to be restricted to the  $O(1)$  region surrounding the tip, while the other regions play a passive role and remains steady. It was found that regardless of the initial condition, a bulbous expanding region forms near the tip in the absence of crystalline anisotropy. For long time, this bulbous region evolves in a way independent of the initial condition. In the presence of noise, represented in our formulation by certain complex singularities, this bulbous tip region will split. The calculation suggests that there can be no globally time dependent state with a locally steady tip. However, with an assumed four-fold crystalline anisotropy, with a minimal surface tension direction coinciding with the needle crystal axis, it is found that the tip region settles to a steady state where the tip radius and velocities are in accordance to microscopic solvability. This happens within the time scale where disturbances have not moved away from the  $O(1)$  region around the tip.

A family of exact solutions describing the time evolution of a zero surface tension dendrite in the  $O(1)$  tip region was reported last year. Some of these solution exist for all times and result in tip-splitting or side-branching of an initially nearly parabolic dendrite. Later on, a criterion involving the sign of the real part of the residues of poles

was found that determined the side of the needle crystal on which disturbances advect. Some of these exact solutions result in a finite-time cusp singularity on the interface. We have addressed the relevance of some of these solutions to the actual evolution of a dendrite in the small surface tension, small Peclet number limit. For instance, through an inner region analysis, it was found that the time at which a zero surface tension solution forms a cusp is preceded by the effect of the so-called daughter singularities that result in tip fattening in the absence of crystalline anisotropy effects. The actual interface in such cases does not come close to cusp formation even for arbitrarily small surface tension. In the presence of anisotropic small surface tension, a cusp does not form either but in this case a locally steady solution results when the anisotropy axis is aligned appropriately.

The fate of various types disturbance advecting along the sides of a needle crystal, as represented by certain types of complex singularities approaching but never hitting the real domain, is also being examined. In particular, it is seen that for an approaching complex pole, significant surface tension effects on the interface occur in a slow time scale that scales inversely with the surface tension parameter. For sufficiently small Peclet number relative to surface tension, such effects can be felt at the interface in the  $O(1)$  region around the tip. For other relative ordering of surface tension and Peclet number, surface tension effects are only felt once the disturbance has advected to other asymptotic regions. The interaction of multiple singularities and their implication to side branching coarsening is currently being examined.

### B. Bridgman Crystal Growth

In the Bridgman problem, we investigated analytically the limit of large solutal Rayleigh number in the case when the rejected solute is heavier than the rest of the alloy, i.e. in a solutally stable arrangement. Unlike the case of large thermal Rayleigh number reported last year, where to the leading order the expressions for radial segregation and crystal melt interface do not involve the heat transfer at the top edge of the insulation zone, we find that in this case the heat transfer at both the top and bottom edges of the insulation zone is involved in the explicit expressions for radial segregation and crystal melt interface shape. We also find some criteria to minimize each of these quantities.

Accurate numerical schemes are being developed to extend and verify the asymptotic scaling results found analytically. In addition, the transient problem that is free of any quasi-steady hypotheses is also being investigated numerically. The numerical results for the quasi-steady equations, so far only with the linearized equations but a more realistic no-slip side wall condition, are in accord with the asymptotic results in the large thermal Rayleigh-number limit, where a more mathematically convenient no-stress condition was used. The calculations also demonstrate clearly the efficacy of the optimal heat transfer condition suggested analytically.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 12/96

PROJECT IDENTIFICATION: 962-24-05-53

NASA CONTRACT NO. NAS1-18605

RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Tanveer, S. Convection effects on radial segregation and crystal-melt interface in vertical bridgman growth. *Phys. Fluids*, 6, 2270-2292 (1994). phase-field models for solidification. *Physica D* 69, 189-200 (1993).

#### Presentations

Tanveer, S. "Crystal growth and fluid mechanics problems in directional solidification." NASA Lewis Workshop, June 1994.

*Oscillatory/Chaotic Thermocapillary Flow Induced by Radiant Heating*

PRINCIPAL INVESTIGATOR: Dr. Robert L. Thompson

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

Prof. K. DeWitt

University of Toledo

Dr. K. Hsieh

NYMA, Inc.

D. Van Zandt

ADF

**TASK OBJECTIVE:**

The main objective of the research is to study the oscillatory and chaotic thermocapillary flows induced by radiant heating of the free surface of a high Prandtl number fluid. Both ground-based experiments and numerical analysis will be conducted to study effect of heating level (supplied by CO<sub>2</sub> laser), surface shape, aspect ratio, and Prandtl number on the conditions for transition from steady to oscillatory flows and then to chaotic flows. In the experiments, flow structures will be observed using a flow visualization technique and temperature distribution on the free surface will be measured using an infrared (IR) imager. Numerical results will be compared with experimental data.

**TASK DESCRIPTION:**

A CO<sub>2</sub> laser is used to provide the heat source. The profile of the laser intensity in the radial direction can be a Heaviside function or a Gaussian function with variable beam diameter. The material of the test chamber is a copper water jacket, providing well-controlled wall temperature. The bottom wall of the test cell is insulated. An IR imager is used to measure surface temperature distribution and several thermocouples are implanted in the test chambers, including one at the bottom. Critical powers of the CO<sub>2</sub> laser at the onset point of oscillatory flow are measured at various aspect ratios and dynamic Bond numbers.

**TASK SIGNIFICANCE:**

The strategy of this study is to first compare the measured onset conditions for oscillatory flows with results from linear stability analysis. With the designed experimental conditions, surface tension and buoyancy effects are equally important. If close comparison between experimental and numerical results can be obtained, it could support the validity of numerical prediction of the onset conditions for pure Marangoni flow. This can help the design of possible space experiments. Results obtained in this study can help the design of surface tension driven convection experiments in space. Furthermore, a deeper understanding of the thermocapillary flow can be achieved, and the knowledge can be used to improve materials processing procedures.

**PROGRESS DURING FY 1994:**

In the study of onset conditions of oscillatory Marangoni flow in laser-heated silicone oil in a cylindrical container, experimental data were obtained for various aspect ratios and dynamic Bond numbers. It is found that for a fixed aspect ratio, there seems to be an asymptotic limit of the dynamic Bond number beyond which no onset of flow oscillation could occur. Experimental results also suggested that there could be a lower limit of the aspect ratio below which there is no onset of oscillatory flow. In parallel, a numerical program for flow instability analysis has been developed to predict the onset of flow oscillations.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 1

PhD Students: 0

TASK INITIATION: 10/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 962-24-05-67

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### **Presentations**

Hsieh, K.C., Thompson, R.L., DeWitt, K., Nash, J. and Van Zandt, D. "Oscillatory/chaotic thermocapillary flow induced by radiant heating." 2nd Microgravity Fluid Physics Conference, Cleveland, OH, June 1994.



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*Light Scattering Studies of Relative Motions of Solid Particles in Turbulent Flows*

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PRINCIPAL INVESTIGATOR: Prof. Penger Tong

Oklahoma State University

CO-INVESTIGATORS:

Prof. W.I. Goldburg

University of Pittsburg

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**TASK OBJECTIVE:**

The primary objective of this task is to understand particle concentration fluctuations in turbulent flows, and in particular how the dispersion is affected by particle inertia and the characteristics of the turbulence.

**TASK DESCRIPTION:**

This proposal describes a series of experiments on the dispersion of particles in turbulent flows. This task plan to study the particle dispersion phenomena in two well characterized turbulent flows. One is turbulent Rayleigh-Benard convection in water and the other is a turbulent grid flow in a water channel.

**TASK SIGNIFICANCE:**

The proposed research will add extremely important data to that presently available to develop understanding of the interaction of solid particles and a turbulent fluid. Such data would be valuable not only in revealing phenomenology, but also in testing model representations of turbulent suspensions.

**PROGRESS DURING FY 1994:**

This is a new grant formally began on July 1, 1994. Necessary advertising and hiring arrangements have been completed to hire a post doctoral associate, who is expected start to work on the project in three months. Meanwhile a new experiment is initiated in Dr. Goldburg's laboratory to study the motion of heavy particles in a turbulent rotating Couette flow. An undergraduate student in Dr. Tong's laboratory is involved in a study of sedimentation of colloidal particles using a newly developed dual-beam incoherent cross-correlation technique.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 7/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-98

RESPONSIBLE CENTER: LeRC

*Computational Studies of Drop Collision and Coalescence*

PRINCIPAL INVESTIGATOR: Prof. Grétar Tryggvason

University of Michigan

CO-INVESTIGATORS:

Dr. D. Jacqmin

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

The objective of this research is to investigate the behavior of bubbles and drops in microgravity by full numerical solutions of the governing equations. The collision and thermal migration of drops are studied in detail to provide essential input for material processing and fluid handling in space. These problems also serve as a test bed for refinements and extensions of the numerical technique being used, thus helping to develop the capability to predict accurately the behavior of free-surface fluid systems.

**TASK DESCRIPTION:**

A numerical technique, based on explicit tracking of the interface between two immiscible fluids, is used in this study. This method has now been extended to deal with both the thermal migration and the rupturing of thin films.

**TASK SIGNIFICANCE:**

The unique aspect of the method is that it accounts fully for both inertia and viscous effects in both fluids and allows the inclusion of surface tension. It is also well suited for complicated interface geometries and has been implemented for fully three-dimensional flows. The basic aspect of this method is described in the *Journal of Computational Physics*, vol. 100 (1992), p. 25.

**PROGRESS DURING FY 1994:**

Our research is aimed at developing a basic understanding of the behavior of drops in microgravity and developing numerical tools to allow accurate predictions of their behavior. We have been focusing mainly on two problems: Collision of drops and thermal migration of both bubbles and drops.

For collision of drops we have simulated the head-on collision of two equal size drops for a wide range of Weber and Reynolds numbers, which are the main controlling parameters, as well as several fully three-dimensional off-axis collisions. The simulations reproduce experimental data well, showing that the evolution of the drops depends to a large degree on the Weber number of the drops before collision, but only weakly on the Reynolds number, once it becomes sufficiently large. One of the more critical questions about drop collisions is whether the drops coalesce permanently or separate again. Several collision modes are possible, and we have examined a number of them. For head-on collisions the drops deform into a disk-like shape as they hit each other and may separate again as surface tension pulls this disk shape back into a more spherical form, often leaving one or more drops in between the original drops. For off-axis collisions, the drops, on the other hand, may continue on their original path after the initial coalescence, and stretch apart again (usually called "grazing collision"). The simulations reproduce these modes well and have clarified a number of points, including the role of dissipation and the role of rupturing of the film between the drops on subsequent evolution.

Our simulations of thermal migration have focused on the collective behavior of many drops as they migrate toward a hot surface. We did several two-dimensional simulations during the first year, to gain experience with our numerical method and to establish a "feel" for the problem. These results showed a strong tendency for many drops to form layers across the channel, and also that the drops only deformed during the initial transient. Recently, the focus has been on the three-dimensional counterpart. These results show that, for a given Ma, the drops do not line up across the channel as rapidly as the two dimensional computations did. Since all interactions fall off much more rapidly in three dimensions as compared with two-dimensions, this is perhaps not too surprising. However,

as the Marangoni number increases, the effect of the drops on the temperature field increases and the interactions become stronger. We are currently investigating the influence of  $Ma$  and other governing parameters, as well as the volume fraction, on these interactions.

During the last year we have also initiated work on the behavior of drops in a shear flow. This allows us to examine the effect of the ambient flow on the drop behavior. We have done an extensive set of two-dimensional simulations and a few three-dimensional ones of the evolution of a layer of drops initially near a wall. The simulations show that as the drops interact the layer breaks up and the drops fluidize as they collide with each other. The rate of fluidization depends strongly on the Weber number which, for this problem, is the ratio of collision time and the time it takes the drops to be advected past each other.

The above computations have all been done using explicit tracking of the boundaries between fluids. Although the method is robust and accurate, it is somewhat complex and for many problems it may be possible to use capturing - where the interface is simply marked by a rapid change in material properties - instead of full tracking. D. Jacqmin has been exploring this possibility and has now developed a code capable of doing short time simulations of relatively complex problems of both coalescence and breakup of drops. For long time simulations, the interface needs to be better preserved, and we are currently exploring ways to do that.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 1/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-24-05-07

RESPONSIBLE CENTER: LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Proceedings**

Heyranni-Nobari, M.R. "Numerical simulations of drop collisions and coalescence." Ph.D. thesis, The University of Michigan, 1993.

**Presentations**

Nas, S. and Tryggvason, G. "Computational investigation of the thermal migration of bubbles and drops." ASME Winter Annual Meeting, 1993.

*Nonlinear Bubble Interactions in Acoustic Pressure Fields*

PRINCIPAL INVESTIGATOR: Prof. John Tsamopoulos

State University of New York, Buffalo

CO-INVESTIGATORS:

Nasser Ashgriz

State University of New York, Buffalo

**TASK OBJECTIVE:**

This project proposes both theoretical and experimental studies of the interactions between bubbles immersed in an immiscible liquid. Specific objectives include:

1. To investigate the conditions when linearly accelerating motion of two interacting bubbles turn into a spiral motion of one of them.
2. To study the delay and prevention of break up of accelerating bubbles.
3. To incorporate bubble velocity and fluid viscosity into a coalescence model.
4. To study the coalescence of two bubbles.

**TASK DESCRIPTION:**

The analytical study will focus on bubble oscillations and interactions of moderately large amplitude and will determine conditions under which the axisymmetric flow turns into three-dimensional. For viscous liquids or large deformation either the Boundary Element method or a combination of Finite Elements with the Volume of Fluid method will be used. Bubbles will be suspended in an immiscible liquid using the acoustic levitation technique for the experimental part of this project. Bubble motions and interactions will be induced by an additional acoustic field. The bubble motions, shape oscillations, collision and breakup will be recorded with high-speed cinematography.

**TASK SIGNIFICANCE:**

The results from this project will be beneficial to others in the bubble dynamics studies. It is well known that the formation and collapse of cavities of gas inside a flow stream cause erosion in fluid-handling equipment. The effect of a properly tuned acoustic field may help in the correct detection of submerged objects in the production of bubble dispersions.

**PROGRESS DURING FY 1994:**

During this reporting period, the following have been accomplished:

1. A cylindrical tank for the acoustic levitation of a single bubble has been designed, built, and tested.
2. A single air bubble has been successfully levitated in the levitation tank filled with water.
3. Shape and volume oscillations of a single bubble is being investigated using a high speed video camera with up to 2000 frames per second.
4. Based on the experimental observations from the single bubble levitator, a new tank is designed which can generate a relatively flat pressure field in order to levitate two or more bubbles. This tank is being built at the present time.

5. A novel numerical technique for the simulation of the interacting bubbles in a viscous fluid is developed. This technique is based on describing the liquid surface by a spine function  $h(a,t)$ , with  $a$  being the angle measured from one axis at time  $t$ . After discretization, the spines  $H_i(a_i, t_i)$  subdivide the liquid zone into conical subvolumes. The volume of each of the subvolumes is updated using the local velocities at the interface of every two neighboring subvolumes. A technique is developed to calculate the new spines based on the updated subvolumes. This method implements Galerkin finite element method with penalty formulation.

6. The new numerical technique has been used to study the oscillation of a single drop and the interaction of two liquid drops. Sample calculations are provided.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-24-05-99

RESPONSIBLE CENTER: LeRC

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## BIBLIOGRAPHIC CITATIONS FOR FY 1994:

**Journals**

Mashayek, F. and Ashgriz, N. Nonlinear instability of liquid jets with thermocapillarity. J. Fluid Mechanics, in press.

Mashayek, F. and Ashgriz, N. Advection of axisymmetric interface. Int. J. Numerical Methods in Fluids, in press.

Mashayek, F. and Ashgriz, N. A hybrid finite element - volume of fluid method for simulating free surface flows and interfaces. Int. J. Numerical Methods in Fluids, in press.

*Residual Accelerations in a Microgravity Environment*

PRINCIPAL INVESTIGATOR: Prof. Jorge Viñals

Florida State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

This research program aims at developing a realistic theoretical model of the high frequency components of the residual acceleration field (or g-jitter), and at studying its effect on a variety of typical fluid experiments.

**TASK DESCRIPTION:**

The high-frequency components of the residual acceleration field are modeled as a stochastic or random process; that is, a succession of random values of the intensity and orientation of the acceleration. Our research is divided into two major parts, one analytic and the other numerical in character. In the first part, we formulate a hydrodynamic problem that explicitly includes a random, time-dependent gravitational acceleration which is modeled as a narrow band noise. In the second part, we develop numerical algorithms to simulate this type of random field and incorporate them into Navier-Stokes equation solvers.

**TASK SIGNIFICANCE:**

All space experiments performed in the manned space environment are exposed to the high frequency components of residual accelerations (g-jitter) on board the spacecraft. This effect can significantly alter experimental results. A numerical approach is taken to determine what regimes of g-jitter will effect experiments conducted in a microgravity environment. A generic class of problems is considered with emphasis on materials experiments.

**PROGRESS DURING FY 1994:**

Buoyancy driven convection induced by a fluctuating acceleration field has been studied in a two dimensional square cavity. This is a simplified model of fluid flow in a directional solidification cell subject to external accelerations, such as those encountered in a typical microgravity environment (g-jitter). The effect of both deterministic and stochastic gravity modulations normal to the initial density gradient are considered. In the latter case, we modeled the acceleration field by narrow band noise defined by a characteristic frequency  $\Omega$ , a correlation time  $t$ , and an intensity  $G^2$ . If the fluid is quiescent at  $t = 0$  when the gravitational field is initiated, the ensemble average of the vorticity at the center of the cavity remains zero for all times. The mean squared vorticity  $\langle \xi^2 \rangle$ , however, is seen to exhibit two distinct regimes: For  $t \ll t_c$ ,  $\langle \xi^2 \rangle$  oscillates in time with frequency  $\Omega$ . For  $t \gg t_c$ ,  $\langle \xi^2 \rangle$  grows linearly in time with an amplitude equal to  $R^2 Pr / (1 + (\Omega t)^2)$ , where  $R$  and  $Pr$  are the Rayleigh and Prandtl numbers of the fluid respectively. At yet later times, viscous dissipation at the walls of the cavity leads to saturation, with  $\langle \xi^2 \rangle_{\text{sat}} = \frac{(Pr\pi + 1)R^2}{(Pr\pi + 1)^2 + \Omega^2 t^2}$ .

A two dimensional model has been introduced to study the onset of parametric surface waves, their secondary instabilities and the transition to spatiotemporal chaos. We obtain the stability boundary of a periodic standing wave state above onset against Eckhaus, zig-zag and transverse amplitude modulations (TAM), as a function of the control parameter  $e$  and the detuning from subharmonic resonance. We find that the Eckhaus and TAM stability boundaries cross at a finite value of  $e$ , thus explaining the finite threshold for the TAM instability observed experimentally. At larger values of  $e$ , a numerical solution of the model reveals the existence of a transition to spatiotemporal chaotic states mediated by the TAM instability. Power spectra of temporal fluctuations in the chaotic state are broadband, decaying as a power law of the frequency  $\omega^{-2}$  with  $z \approx 4.0$ .

Finally, we have started the analysis of g-jitter data gathered by the SAMS project during the SL-J Mission. Of particular concern is the detection of deterministic and stochastic components, and a comparison of the latter with the stochastic model of g-jitter currently used.

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STUDENTS FUNDED UNDER RESEARCH:				TASK INITIATION:	6/91	EXPIRATION:	9/95
BS Students:	0	BS Degrees:	0	PROJECT IDENTIFICATION:	962-24-05-36		
MS Students:	0	MS Degrees:	0	NASA CONTRACT NO.:	NAG3-1284		
PhD Students:	2	PhD Degrees:	1	RESPONSIBLE CENTER:	LeRC		

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

#### Journals

Casademunt, J., Zhang, W., Vinals, J., and Sekerka, R.F. Stability of a fluid surface in a microgravity environment. *AIAA J.*, Vol. 31, 2027 (1993).

Chella, R. and Vinals, J. Order parameter representation for the motion of an active interface during mixing in cavity flow. *AIAA Journal*, (preprint).

Gurtin, M., Polignone, D. and Vinals, J. Two-phase binary fluids and immiscible fluids described by an order parameter. *J. Fluid Mech.*, (preprint).

Thomson, J.R., and Vinals, J. Statistical saturation of buoyant flow induced by a fluctuating acceleration. *AIAA Journal*, (preprint).

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Zhang, W. and Vinals, J. Secondary instabilities and spatiotemporal chaos in parametric surface waves. *Phys. Rev. Lett.*, (in press).

Zhang, W., Casamunt, J., and Vinals, J. Study of the parametric oscillator driven by narrow band noise to model the response of a fluid surface to time-dependent accelerations. *Phys. Fluids A.*, Vol. 5, 3147 (1993).

#### Proceedings

Thomson, J.R. and Vinals, J. "Cavity flow induced by an effective random gravitational field." *Proceedings of the 32nd Aerospace Sciences Meeting*, Paper AIAA-94-0438, 1994.

#### Books

Casademunt, J. and Vinals, J. "Convection induced by a fluctuating acceleration field," in "Fluid Mechanics in Microgravity." Edited by: D.A. Siginer, R.L. Thomson, and L.M. Trefethen ASME, New York, NY, pp 133. 1993.

#### Presentations

Vinals, J. "Residual accelerations in a microgravity environment." *Second Microgravity Fluid Physics Conference*, Cleveland, OH, June 1994. NASA Conference Publication 3276.

*Studies of the Dynamics of Charged Free Drops*

PRINCIPAL INVESTIGATOR: Prof. Taylor G. Wang

Vanderbilt University

CO-INVESTIGATORS:

C.P. Lee

Vanderbilt University

A.V. Anilkumar

Vanderbilt University

**TASK OBJECTIVE:**

The dynamic behavior of a charged liquid drop has been the subject of investigation. The basic assumption is that the charge essentially reduces the surface tension of a neutral drop; hence the shapes and stabilities of a liquid drop are greatly affected by the amount of charge presented on the drop surface. Various theories have been developed to predict the dynamic responses of a drop under different charge conditions.

**TASK DESCRIPTION:**

One of the major technical problems facing all charged-drop experiments is the determination of charge quantity on a liquid sample. To avoid the contamination and source loading problems, a noncontact charge-measuring technique is preferred. From basic physics, there is only one way to determine the potential of a charged object by noncontact means, and that is to measure the strength of the electrostatic field generated by the charge object. With precise calibration based on the geometry of the system and measuring distance, the field strength can be used to obtain the potential of the charged object.

**TASK SIGNIFICANCE:**

The study of the oscillation and rotational dynamics of charged liquid drops sustained by surface tension is interesting from both a fundamental standpoint and a practical standpoint vis-a-vis processing materials in space. The proposed ground-based experiments will help us to further the understanding of drop behavior and to optimize the approved flight experiments.

**PROGRESS DURING FY 1994:**

**Charged Drop Task**

A charged-drop positioning device has been fabricated which allows charge measurement of the small volumes of liquid which can be levitated on the ground. The average charge density is measured; the charge distribution is not controlled. The final phase of this task has focused on studying the dynamics of charged drops undergoing large-amplitude oscillations. High-speed imaging has been used to record the drops' behavior.

**DPM Technical Support Task**

The most recent and final work requested by the Drop Physics Module (DPM) Project has been to identify the cause of the anomalous rotation observed in the DPM during USML-1. A flow-visualization system was built up to study the flows inside acoustic chambers using both a DPM-like chamber as well as one with a simpler geometry; The behavior of the smoke was analyzed with isolated acoustic signals, with the complicated mix of signals used on USML-1, and with the USML-2 signals designed to control the tumble rotation.

A very long fiber was used to measure the actual torque on a spherical target in the acoustic chamber under the conditions which caused flows in the earlier tests. After extensive searching a very limp fiber was found so that the sample and fiber could be mounted on a fixture which could make similar torque measurements in the flight chamber of the DPM. Measurements were made in that chamber while the DPM was at KSC as well as in the chamber in the trainer at the Payload Crew Training Complex. The results of these tests are being analyzed and will be documented in a final report by December 1994.



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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
 MS Students: 0  
 PhD Students: 2

TASK INITIATION: 1/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-24-07-09

RESPONSIBLE CENTER: JPL

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**Journals**

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**Proceedings**

Hmelo, A.B., Grugel, R.N., Wang, T.G. "Review of experimental facilities and reduced gravity platforms of containerless experimentation." Proceedings of the TMS meeting, Denver, CO, 1993.

*Experimental Study of the Vapor Bubble Thermosyphon*

PRINCIPAL INVESTIGATOR: Prof. Peter C. Wayner, Jr.

Rensselaer Polytechnic Institute

CO-INVESTIGATORS:

Prof. J. Plawsky

Rensselaer Polytechnic Institute

**TASK OBJECTIVE:**

The objective of this effort is to better understand the physics of evaporation and condensation as they affect the heat transfer processes in a vapor bubble thermosyphon (VBT). In small systems, interfacial intermolecular forces can be used to control fluid flow and heat transfer. The VBT, one such system, consists of a small enclosed container partially filled with a liquid. When a temperature difference is applied to the ends of the VBT, evaporation occurs at the hot end and condensation at the cold end -- resulting in a very effective heat transfer device.

**TASK DESCRIPTION:**

A transparent VBT will be designed and developed. The microscopic intermolecular force (pressure) field, which is a function of the liquid thickness profile, will be measured using microcomputer enhanced video microscopy based on interferometry. The temperature field will be measured using the interline absorbed film thickness and small temperature sensors.

Models of the transport processes in the contact line region of a VBT which include the effects of liquid-solid and liquid-vapor intermolecular forces have already been developed. As part of this effort these models will be further refined and the transport characteristics of VBTs will be obtained by comparing the experimental data to numerical solutions of the model.

**TASK SIGNIFICANCE:**

By studying liquid-film thicknesses and temperatures in VBT, a better understanding of the processes can be gained that will lead to optimization of TBV designs.

**PROGRESS DURING FY 1994:**

A preliminary experimental study of vapor bubble thermosyphons has been accomplished. In it measurements of wall temperatures (using thermocouples) and liquid film thicknesses (using an image analyzing interferometer) were made to characterize behavior of the VBT; valuable experience was gained in the operation of such systems. Difficulties were encountered in finding a proper fluid and container material combination so that the system will have desirable characteristics both optically and thermally. Proper procedures for filling the cells have been found to be important in avoiding contamination. VBT cells for the main experiment have been designed and are currently being constructed.

The experimental studies can be divided into a equilibrium and non equilibrium studies of the VBT. The equilibrium studies have progressed to the point where we can make the following conclusions:

1. The use of an image analyzing interferometer, IAI, with a constrained vapor bubble thermosyphon, (CVBT), was demonstrated under equilibrium conditions at 1g.
2. Using the augmented Young-Laplace equation good agreement between the theoretical and experimental values of the dispersion constant was obtained.

3. A gravitational field restricts the range of forces that can be studied because the curvature gradient,  $K'$ , is a function of gravity.
4. Ground based studies indicate that a flight experiment can be designed to evaluate the augmented equilibrium Young-Laplace equation using the CVBT.
5. A flight experiment for (at least) a study of the augmented equilibrium Young-Laplace equation is warranted.
6. Ground based non equilibrium experiments are being developed.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-05-44

RESPONSIBLE CENTER: LeRC

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DasGupta, S., Plawsky, J.L., and Wayner, Jr. P.C. "Determination of the Hamaker Constant in an extended meniscus using image processing." Abstract accepted for presentation at the 1994 annual meeting of A.I.Ch.E., San Francisco, CA. Nov. 13-18, 1994.

*Interactions Between Solidification and Compositional Convection in Alloys*

PRINCIPAL INVESTIGATOR: Prof. M. G. Worster

Northwestern University

CO-INVESTIGATORS:

Prof. S.H. Davis

Northwestern University

**TASK OBJECTIVE:**

The project aims to quantify the effects of convection on the structure and composition of cast alloys. Particular attention will be focused on the form and influence of convective flows through the interstices of mushy layers during solidification.

**TASK DESCRIPTION:**

Combined experimental and theoretical studies will be undertaken. The laboratory experiments will involve the solidification of aqueous salt solutions, as representatives of general binary systems. The theoretical studies will employ linear and nonlinear stability theory, asymptotic and numerical methods in the development and analysis of predictive mathematical models.

**TASK SIGNIFICANCE:**

This study of fluid dynamics during solidification will aid the design of improved casting procedures (e.g., for the manufacture of high-performance turbine blades). Additionally it will improve our understanding of air-sea interactions in polar regions, where the formation of sea ice is a dominant contributor to the global heat budget, leading to better climate modeling and prediction.

**PROGRESS DURING FY 1994:**

Significant progress has been made with a study of the evolution of nonlinear perturbations to a growing mushy layer. In addition to determining the stability of various finite-amplitude convective states, we have determined the physical mechanisms that control the degree of subcriticality of the bifurcation's to convection within a mushy layer. We have discovered a hitherto unsuspected oscillatory mode of instability in the mushy layer that is a consequence of the intimate coupling between solidification and convection, and may cause compositional striations to occur in a cast alloy.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-24-05-63

NASA CONTRACT NO.: NAG3-1405

RESPONSIBLE CENTER: LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:**

**Journals**

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*Nucleation and Chiral Symmetry Breaking under Hydrodynamic Flows*

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PRINCIPAL INVESTIGATOR: Dr. Xiao-lun Wu

University of Pittsburgh

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The aim of this research is to investigate nucleation and chiral symmetry breaking under shear flow. It has been proposed that the symmetry breaking is a result of a strong first order phase transition coupled to a macroscopic, velocity field. The process, often referred to as autocatalysis, is considered as one of the key steps for chemical and biological evolutions. However, its very existence remains elusive.

**TASK DESCRIPTION:**

An individual molecule of sodium chlorate ( $\text{NaClO}_3$ ) is symmetric, but its crystal form shows distinctive optical activity. If a solution of sodium chlorate is left undisturbed, hundreds of crystals slowly form as the solvent evaporates. If these crystal are examined under crossed polarizers, one finds that half are left-handed and the other half are right-handed. Equal percentage of the two species indicates that the energy barrier for the nucleation is the same for the different handedness. It was discovered by Kondepudi et al. that under gentle stirring condition, the crystallization seems to proceed in one direction, favoring either all crystals being right-handed or left-handed for each individual experiment. The optical purity in each experiment is greater than 99%. However, if an ensemble of experiments is carried out under the same conditions, one again finds an equal distribution of left and right-handed species. This remarkable observation indicates that the system undergoes a transition from a totally symmetric state to a totally asymmetric state simply by the introduction of a hydrodynamic flow.

The current experiments are designed to probe two important aspects of the above observations. (1) We would like to know if the transition from the symmetric state to the asymmetric state is an abrupt transition or a smooth one, as the flow parameters are varied. (2) To observe the existence of autocatalysis it will be useful to find correlations between the local order parameter  $O = (n_L - n_R) / (n_L + n_R)$ , averaging over some spatial scale  $l$ , and hydrodynamic flow patterns in the system. Here  $n_L$  and  $n_R$  are the number densities of left and right handed crystals on the scale  $l$ . Low Reynolds number hydrodynamic convections, such as Taylor-Couette flow and Reyleigh-Benard convections provide fertile testing grounds. In these hydrodynamic systems, the flow consists many local coherent structures, rolls and eddies, and the convective mixing is most effective within these coherent structures.

**TASK SIGNIFICANCE:**

Effect of hydrodynamic convection on phase transitions is a significant problem not only for basic science but also for technology. Microgravity provides a unique environment for which some of the above theoretical ideas can be tested. Here minimization of sedimentation is crucial for both the experimental observations and for the theoretical analysis. This study will enrich our general knowledge about nucleation and growth under hydrodynamic flow and may be exploited to produce chirally pure materials.

**PROGRESS DURING FY 1994:**

To investigate the sharpness of the transition we have built a mixing apparatus which accommodates 20 samples at a time. The flow in the samples is created by small magnetic stirring bars driven by horse-shoe magnets. To ensure uniform flow for all the samples, the horse-shoe magnets are connected in tandem, driven by a DC motor. We have incorporated a microscope and a video imaging system for counting the crystals when they grow to a fraction of a millimeter in size. Our preliminary results suggest that the transition is rather smooth as the stirring frequency is varied. The critical frequency  $f_c$  appears to be around 2 Hz. For  $f < f_c$ , the chiral symmetry is only partially broken, whereas for  $f > f_c$ , the symmetry breaking is almost a certainty (with probability greater than 90% for individual

runs). More recently we discovered that the critical frequency  $f_c$  seems to depend on the volume of the solution used, and possibly also depends on the location of the magnetic stirring bar. This new finding may be important in differentiating the effects of hydrodynamic convection and mechanical damage of incipient nucleus. It remains an intriguing possibility that both processes may be responsible for the chiral symmetry breaking transition.

To correlate nucleation events with the underlying hydrodynamic convection patterns, we have constructed an electro-convection cell. The solution, containing 50% solid weight fraction of  $\text{NaClO}_3$ , is a weak electrolyte. When a thin layer of  $\text{NaClO}_3$  solution is placed above an array of alternating magnets and driven by a DC current, the fluid layer forms a set of vortices, which is commensurate with the arrangement of the magnets underneath the fluid. The nucleation experiment under electro-convection is currently underway and its result will be reported elsewhere.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/93 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-24-08-12

NASA CONTRACT No.: NAG8-959

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Wu, X.-L., Martin, B., and Tharrington, A. "Nucleation and chiral symmetry breaking under hydrodynamic flows." NASA Microgravity Fluids Conference, Cleveland, OH, 1994.

*Oscillatory Thermocapillary Convection*

PRINCIPAL INVESTIGATOR: Prof. Abdelfattah Zebib

Rutgers University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The main objective of this work is to investigate the influence of free-surface deflection on the onset of oscillatory thermocapillary convection in microgravity. The study is to investigate the flow instability of thermocapillary flow. The Hopf bifurcation will be studied using a numerical method with the consideration of free-surface deflection. In addition, the disappearance of the Hopf bifurcation will be investigated.

**TASK DESCRIPTION:**

In the domain perturbation approach (valid for small capillary numbers ( $Ca \rightarrow 0$ )), time-dependent, two-dimensional combined buoyant thermocapillary motions in a rectangular cavity are computed using a second-order accurate finite-volume method. Two situations are investigated: The  $O(1)$  pure buoyant convection (with the Marangoni number  $Ma=0$ ) is known to exhibit a Hopf bifurcation at some critical value of the Grashof number,  $Gr_{cr}$ . Thus, by studying the combined thermocapillary-buoyant convection for values of

$$(Ma, Gr) \text{ near } (0, Gr_{cr})$$

we seek to determine the stability boundary for onset of oscillatory motion and its nature in the limit of vanishing buoyancy,  $Gr=0$ . Both positive and negative values of  $Ma$  are considered. In the second problem we consider pure thermocapillary driven convection ( $Gr=0$ ). The solution to the zeroth order system is known to be stationary. Higher order effects are unknown and will be investigated.

**TASK SIGNIFICANCE:**

These studies can contribute to our fundamental understanding and potential control of thermocapillary instabilities. Detailed information of thermocapillary flows can be obtained through this comprehensive numerical study. Mechanisms for controlling the flows will be identified. Thus the fluid management and heat transfer processes will be better controlled in space.

**PROGRESS DURING FY 1994:**

For low Prandtl number fluids, the solution to the zeroth order system is known to be stationary at parameter values of interest in semiconductor crystal growth. We have established that incorporating higher order terms also leads to steady thermocapillary motions. This important result is both in agreement and disagreement with recently published papers by other researchers employing different computational methods. Results from current study showed that there exists a critical Rayleigh number below which no flow oscillations could occur. Different conclusions were drawn from other researchers in the studies of similar configurations.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-24-05-49

RESPONSIBLE CENTER: LeRC

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Mundrane, M., Xu, J., and Zebib, A. Thermocapillary convection in a rectangular cavity with a deformable interface. *Advances in Space Research*, in press.

**Presentations**

Mundrane, M. and Zebib, A. "Oscillatory thermocapillary convection." Second Microgravity Fluid Physics Conference, Cleveland, OH, June, 1994.

Mundrane, M. and Zebib, A. "Oscillatory, two-dimensional buoyant thermocapillary convection." ASME-WAM, New Orleans, LA, November, 1993.

Mundrane, M. and Zebib, A. "Marangoni convection with a deformable interface." ASME-IMECE, Chicago, IL, November, 1994.

Mundrane, M., Xu, J., and Zebib, A. "Thermocapillary convection in a rectangular cavity with a deformable interface." COSPAR '94-G1-Symposium, Hamburg, German, July 12-16, 1994.

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*Synthesis and Characterization of Single Macromolecules: Mechanistic Studies of Crystallization and Aggregation*

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PRINCIPAL INVESTIGATOR: Prof. Spiro D. Alexandratos

University of Tennessee

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## CO-INVESTIGATORS:

K. Cook

University of Tennessee

D. Joy

University of Tennessee

P. Phillips

University of Tennessee

B. Wunderlich

University of Tennessee

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**TASK OBJECTIVE:**

The principal objective is to prepare single-molecule polymer amorphous particles and crystals by thermally induced precipitation / crystallization from bulk dilute solutions and by precipitation / crystallization via solvent evaporation from electrosprayed nanodroplets of dilute solutions. Polymer-polymer interactions will be controlled in order compare the effects of interchain and intrachain entanglements on the chemical and physical properties of a polymer particle or crystal. Successful preliminary studies will be extended to studies in microgravity which will allow for a much more sensitive probing of intermolecular interactions through differences in properties of the polymers.

**TASK DESCRIPTION:**

An electrospray attachment will be constructed for a conventional mass spectrometer, allowing for individual polymer chains to be isolated as they emerge from the instrument. Gravity complicates their isolation but its effect could be minimized with very dilute solutions. The morphology of the single chains will be characterized by high resolution low voltage scanning electron microscopy. Backscattered electron imaging will also be utilized, with spatial resolution of 1-3 nm. Thermal analysis will reveal details of chain motion, including details of crystallization kinetics and the effects of inter- and intrachain entanglement.

**TASK SIGNIFICANCE:**

The commercial and technological importance of plastics cannot be overstated. Specially designed plastics have been applied as materials in aerospace construction, medical prosthetics, and organic semiconductors. This wide range of applications stems from the wide diversity of chemical and physical properties of the macromolecules (i.e., polymers) which serve as the building blocks of the plastics we encounter. While our understanding of the origins of this diversity has advanced in recent years, it remains far from perfect. With the increasing importance of composite materials, the gap between theory and experiment may even be widening. An investigation into the role of the interactions between discrete macromolecules on the final observed macroproperties requires the availability of samples of controlled molecularity (i.e., one, two, three,... macromolecules). It is the goal of this research to prepare and characterize samples comprised of single polymer molecules of known molecular weight, then compare the observed properties with those of comparably well-controlled systems where the number of interacting macromolecules is known. This will allow for the design of new plastics with targeted properties including strength, durability, and biodegradability.

**PROGRESS DURING FY1994:**

This research had September 1, 1994 as its official beginning. We have defined a series of experiments and have identified a post-doctoral research associate who will be responsible for carrying out the experiments. The post-doctoral associate has recently accepted our offer. Laboratory space has been identified and the necessary supplies are being purchased.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-21-08-25

NASA CONTRACT NO.: NAG8-1065

RESPONSIBLE CENTER: MSFC

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*A Novel Electrochemical Method for Flow Visualization*

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PRINCIPAL INVESTIGATOR: Dr. T. J. Anderson

University of Florida

## CO-INVESTIGATORS:

R. Narayanan

University of Florida

A. Fripp

NASA Langley Research Center (LaRC)

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**TASK OBJECTIVE:**

The objective of this research program is to develop and demonstrate a novel electrochemical technique to visualize the dynamic states in high temperature liquid metals. This technique uses the oxygen anion-conducting ceramic electrolyte yttria stabilized zirconia in a crucible material which holds the model liquid metal tin in a Bridgman configuration. Electrochemical cells will be constructed at various positions along the side wall and bottom of the solid electrolyte. The bottom cell will operate in the electrolytic mode to establish a well defined boundary condition with respect to the titrated oxygen tracer concentration. The side wall cells will be operated in the galvanic mode to measure the dynamic oxygen concentration at the various wall locations. Well defined convective flow patterns will be established for this model geometry and the results will be compared to numerical predictions of the flow patterns. In this manner the sensitivity of the technique will be determined.

**TASK DESCRIPTION:**

In order to identify the sensitivity of the technique, a cylindrical crucible with local electrochemical cells at 5 vertical and 4 azimuthal positions (20 total cells) will be constructed. A thin (1/8") alumina rod will be rotated in the melt along the central axis in an isothermal furnace to produce a well defined dynamic state. At suitable rotation speed and aspect ratio, Taylor vortices are expected and the oxygen concentration variation along the side wall will be measured after establishing a zero concentration boundary condition at the bottom face of the melt cylinder. Next, the flow directions of dynamic states (oscillatory flow) developed in a Sn melt without the rod in a destabilizing vertical temperature gradient will be studied. In this experiment, a single cell will be electrochemically pulsed and the time response monitored at the other cells. The experiment will be repeated with different electrolytic cells and pulsing experiments will also be performed to identify the natural oscillation frequency. In a final series of experiments, the continuous response of the sensor cells will be measured to a step change in boundary condition. The results will be compared to a complete 3 D calculation of buoyancy driven flow accounting for sidewall conduction and radial transport.

**TASK SIGNIFICANCE:**

In the processing of many advanced materials, for example the bulk crystal growth of semiconductors, the fluid flow state of the liquid determines the quality of the material produced. Although flows in the liquid are not intentionally created, natural forces produce flows. Since the melt is often at high temperature, exhibits a high vapor pressure, and is not transparent, it is extremely difficult to see the fluid flow pattern. Thus, we must rely on the results of calculations which have drawbacks. We have proposed a novel method to measure flow patterns in liquid metals by using solid state electrochemical sensors to measure the time variation of a tracer species. In a microgravity environment, it is anticipated that the magnitude of natural fluid flow will be greatly reduced and produce improved materials. In order to correlate material properties to the fluid flow pattern, we must know the pattern. This technique promises to provide such insight. Potentially, the method could be adapted for improved process control and manufacturability.

**PROGRESS DURING FY 1994:**

This project has been active for 4 months. In this time, the furnace system, gas purification manifold and electrochemical measurement equipment have been established. An alumina based crucible system will be necessary to avoid wall transport of the oxygen tracer. A design of the crucible has been developed which incorporates tapered

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

yttria (YSZ) stabilized zirconia plugs located along the sidewall and a YSZ disc at the bottom. Work is in progress to incorporate computer based data acquisition. In addition, 3 D modeling efforts of the Bridgman geometry have been initiated. A student has been recruited from the Fall entering class and a post doc with experience with solid electrolytes will join the program in November.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	2	BS Degrees:	1
MS Students:	0	MS Degrees:	0
PhD Students:	0	PhD Degrees:	0

TASK INITIATION: 7/94    EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-21-08-26

NASA CONTRACT No.: NCC8-51

RESPONSIBLE CENTER: MSFC

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*Foam Metallic Glasses*

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PRINCIPAL INVESTIGATOR: Prof. Robert E. ApfelYale University

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## CO-INVESTIGATORS:

N. Qiu

Yale University

C. Boa-Teh

Yale University

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**TASK OBJECTIVE:**

The scientific objective is to determine the parameter space for which foamed glasses are possible. The engineering objectives are to design apparatus to operate in this parameter space and to investigate the practicality and desirability of this process for satisfying technological needs and for producing new opportunities for the application of these materials.

**TASK DESCRIPTION:**

Amorphous materials are an important class of materials because of their many unique features, such as the absence of crystal defects (e.g. grain boundaries or dislocations) and a wide compositional range over which a single-phase amorphous material can be formed. A novel processing approach and experimental design to achieve as-cast bulk amorphous materials are investigated, since a bulk form of amorphous material is required in many applications. By sudden decompression of a melt that is seeded with a volatile liquid, the dispersed "foaming" liquid vaporizes, taking its latent heat of vaporization from the melt, thereby homogeneously cooling the melt. Due to a high decompression rate, a sufficient cooling rate may be produced to yield an amorphous solid foam.

The experimental program is divided into three major sections: proof of foaming principle with organic materials; tests with the water-tin system, even though it is known that such systems will not form a glass; and tests of an alloy system for forming a foamed metallic glass. The first of these is to verify our expectations with regard to the foaming process and the production of a bulk foam. The second is to give us experience with a metallic system that others have worked with and which may present behaviors unique to metals and not observed with organic materials. The third step is obviously an important milestone toward foamed metallic glasses. The experimental program will be complemented by a theoretical/computational study of this highly transient "foaming" process and by comprehensive materials analysis of all product specimens.

**TASK SIGNIFICANCE:**

Rapid decompression of seeded melts is a novel processing approach to produce foam metallic glass which is an open solid bulk structure that may have glass properties and low density. These foam metallic glass materials should possess few structural defects and may have many potential applications, such as lightweight and high strength structural materials, or matrix materials that can be filled with other materials to suit some special requirements.

**PROGRESS DURING FY 1994:**

Preliminary results have shown that this novel method for solid foam processing is feasible. Foam p-terphenyl has been produced by sudden pressure decompression of melted p-terphenyl in which water is dispersed. The foam is an open, porous and yet interconnected structure, and can be made with a density as low as 12% of the original density of p-terphenyl. High water to p-terphenyl ratio and high releasing pressure help produce foam with high porosity or low density.

Limited experiments of foam processing of metal tin, using the same apparatus for p-terphenyl, and water as the foaming agent, were performed. Within the conducted experimental regime, no massive foam was observed and the resulting tin was not an open structure. However, in some surface areas, holes and convex hills and dimples were observed. Some features are similar to those of foam p-terphenyl. The differences of surface tension and density

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

between tin and water are large. Tin and water tend to separate rather than mix together even at elevated temperature. Construction of a more sophisticated apparatus is currently in progress in order to disperse water uniformly into tin to improve the chances of producing foam tin. In addition to the experimental progress, the initial formulation of theoretical/computational study of this highly transient "foaming" process has been made.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 3/93 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-25-08-24

NASA CONTRACT No.: NAG8-947

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Apfel, R.E., and Qiu, N. "Foam metallic glass." Proceedings of the NASA Microgravity Materials Science Conference, Huntsville, AL, 129 (1994).

*Nucleation and Cluster Formation in Levitated Droplets*

PRINCIPAL INVESTIGATOR: Prof. Stephen Arnold

Polytechnic University, New York

## CO-INVESTIGATORS:

A.F. Izmailo

Polytechnic University, New York

A.S. Myerson

Polytechnic University, New York

**TASK OBJECTIVE:**

1. Employment and improvement of the existing experimental Electrodynamic Levitator Trap (ELT) technique in order to investigate stochastic motion of the ELT confined microdroplets of supersaturated solutions in the case where the dimensionless drag  $a$  and driving  $b$  parameters are much greater than one;
2. Development of an appropriate theoretical formalism to describe stochastic motion of the ELT confined microdroplets of supersaturated solutions in an atmosphere near the Standard Temperature and Pressure for the case where the dimensionless drag  $a$  and driving  $b$  parameters are not necessarily small (less than one).

**TASK DESCRIPTION:**

Components of this research include:

1. Experimental study of stochastic motion of the confined levitated microdroplets of various supersaturated solutions. The containerless levitation allows to considerably delay nucleation onset and, thus, provides an unique method to study the formation and evolution of subcritical solute clusters. This study includes measurement of such time-dependent microparticle characteristics as the standard deviation of its confined stochastic motion, activity of the solute dissolved, etc.. Since the measured characteristics are extremely sensitive to the appearance of subcritical solute clusters (solid inclusions) inside of the studied microdroplet the proposed experiment provides an unique opportunity to study metastable state of matter.
2. Development of theoretical models of the microparticle confined stochastic motion for the two principally different cases: (1) when there are no solid inclusions inside, i.e., when the microdroplet solution is undersaturated, and (2) when there are solid inclusions inside of the microparticle, i.e., when the microdroplet solution is supersaturated. These two models will be developed for the particular case where energy dissipation of the microdroplet motion in the levitator atmosphere is linear.

**TASK SIGNIFICANCE:**

Understanding of the metastable state evolution in supersaturated solutions is of extreme importance in the problem of governed nucleation and crystal growth. Since this evolution consists of the birth-death process of subcritical solute clusters, we outline the following significance:

1. It is a challenging experimental problem to study metastability since any heterogeneity may cause instantaneous nucleation followed by crystallization. Therefore, development of the containerless (without heterogeneities due to container walls) experimental technique is of interest.
2. Analytical description of stochastic motion of the confined supersaturated solution microdroplet will allow treatment of the obtained experimental data.

**PROGRESS DURING FY 1994:**

We have performed experiments with microdroplets of various electrolyte and non-electrolyte supersaturated solutions. The obtained experimental results and their theoretical treatment for the case of electrolyte supersaturated

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

solutions have allowed to make some interesting conclusions concerning distribution of subcritical solute clusters. It appears that there is such a particular solute supersaturation concentration when solution electrical conductivity becomes equal to zero, i.e., all solute ions dissolved become associated into electrically neutral Bejrrum pairs.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-25-08-30

NASA CONTRACT No.: NAG8-1060

RESPONSIBLE CENTER: MSFC

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Izmailov, A.F., Arnold, S., and Myerson, A.S. Parametrically driven microparticle in the presence of a stationary zero-mean stochastic source: model for thermal equilibrium in the Paul trap. Phys. Rev. E, vol. 50, 702 (1994).



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*Microwave Materials Processing in Microgravity*

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PRINCIPAL INVESTIGATOR: Dr. Martin B. Barmatz

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The Microwave Materials Processing in Microgravity task objective is to apply the unique capabilities of microwave heating and positioning to process materials in microgravity. The task objectives will include 1) determination of the reaction mechanism, microstructure development and physical properties associated with microwave synthesis of ceramics, 2) development and application of microwave techniques for (a) monitoring the energy absorption during processing and (b) measuring thermophysical properties of materials in microgravity, and 3) theoretical modeling of unique microwave heating and positioning capabilities in a microgravity environment.

**TASK DESCRIPTION:**

There is a recognized need to produce advanced refractory ceramics that have higher melting temperatures and improved mechanical properties (such as strength and toughness). In recent years, ground-based experiments using microwave heating have demonstrated enhanced rates of sintering of ceramic materials leading to new microstructures. The synthesis of ceramics in a microgravity environment could provide the opportunity to produce contamination free ceramics with controlled microstructures that lead to advanced structural applications. Microwave processing can heat many glass and ceramic compositions very rapidly to high temperatures, it can heat them more uniformly than other methods, and it is energy efficient. Of particular interest is the application of microwave to the combustion synthesis process. Other important potential applications are crystal growth, and fiber pulling in space. Microwaves can generate a well defined temperature gradient within a material leading to the possibility of melting only the interior of a cylindrical sample, or leading to a radial gradient of the index of refraction upon solidification. By appropriate monitoring of the microwave parameters during processing one can also measure various sample properties as well as obtain energy absorption information which can be used to characterize the sample reaction and densification mechanisms. Microwaves can also produce unique positioning forces.

**TASK SIGNIFICANCE:**

This task may lead to a new novel technique for the efficient processing of high temperature ceramics in a microgravity environment. The ability to microwave heat and position a sample may also lead to a new containerless technology that is ideally suited for controlled processing of materials in microgravity.

**PROGRESS DURING FY 1994:**

During the last year, we have (a) developed a new plasma enhanced microwave joining technique. This method was used to join 99.8% pure alumina rods at 2000 C, (b) successfully demonstrated microwave annealing of Cu thin films on silicon substrates to eliminate the voids present in sub-micron size interconnects. This new technique could support the development of ultra large-scale integrated (ULSI) semiconductor circuits, and (c) demonstrated microwave ignition of the combustion synthesis process to produce dense high strength TiC.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 10/81 EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-25-04-07

RESPONSIBLE CENTER: JPL

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Brain, R.A., H. A. Atwater, and M. Barmatz. "Rapid selective annealing of Cu thin films on Si using microwaves." *MRS Spring Symposium Proceedings*, 1994.

*Studies of Containerless Processing of Selected Alloys*

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PRINCIPAL INVESTIGATOR: Prof. Robert J. BayuzickVanderbilt University

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## CO-INVESTIGATORS:

W. Hofmeister

Vanderbilt University

M. Robinson

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

The research focuses on determining the speed of solidification as a function of undercooling in the large undercooling regime.

**TASK DESCRIPTION:**

The work involves the undercooling of refractory metals and alloys by containerless processing. In particular, experiments are done by use of electromagnetic levitation (on the bench) at Vanderbilt University. Specifics addressed are maximum undercoolings in refractory metals and alloys, nucleation frequency in refractory metals and alloys, the effect of undercooling on solidification velocity in refractory metals and alloys, and microstructural development in refractory metals and alloys as a function of deep undercooling/solidification velocity and rapid heat removal.

This work consists of a comprehensive investigation on growth of the solid from the undercooled liquid in selected metals and alloys. In order to do this, containerless processing is essential not only to eliminate the effect of the container on nucleation but also because the alloys of interest are highly reactive and become contaminated with crucible material.

The ground-based technique for containerless processing applied to this effort is electromagnetic levitation on the bench at Vanderbilt University.

The alloys studied are TiAl based. In addition, studies are done on pure Ti and pure Ni, as points of reference and because of their stand alone contribution to science. The particular alloys are chosen because they exhibit a wide solubility range as seen in the phase diagram and, within compositions ranges, exhibit a tendency to be unencumbered by competition between alternate phase selection in the deep undercooling regime.

The alloys as well as pure Ti and Ni are studied in order to determine the solidification velocity as a function of melt undercooling. The velocity is determined using ultra high speed imaging to track the solidification front as it moves through the specimen. In opaque samples this can be done by monitoring the movement of the thermal field developed by recalescence. An instrument has been developed that consists of a 10 by 10 array of parallel tapped photodiodes and data acquisition. The system is capable of capturing data at rates of up to 1 million frames per second and the output from the array is calibrated for conversion to temperature. For this alloy and each pure element, a plot of the solidification velocity as a function of undercooling is developed. Of particular interest are the mechanisms controlling the rate of solidification at various undercoolings.

**TASK SIGNIFICANCE:**

This research is of interest not only because of its contribution to solidification theory but also because of its relevance to the development of useful high temperature alloys. In order to understand the mechanical properties of these high-temperature materials the microstructure of the material must be understood. This requires an understanding of all of the factors that contribute to the genesis of the microstructure. In particular, the velocity with which the solid grows into the liquid is pertinent.

From a purely scientific stand point the relationship of the solidification velocity to the melt undercooling is not well understood. The data reported by various scientists conflict with each other and are not in good agreement with solidification theory. It is, therefore, necessary to build a quantitative experimental data base that can be compared to theory and allow the areas of discrepancy to be addressed.

**PROGRESS DURING FY 1994:**

The measurement of the solidification velocity of undercooled liquids has continued with the development of an ultra high speed imaging system. The system consists of a parallel tapped ten by ten array of photodiodes, amplification stages and data acquisition. The system captures data at rates ranging from 500 to 1,000,000 frames per second and the output is calibrated for temperature measurement.

The solidification velocity of Ti-60a/o Al was measured using the ultra high speed imaging system. Equilibrium solidification of Ti-60a/o Al produces the ordered intermetallic gamma phase with the L10 structure. Theory predicts that this alloy will solidify in the ordered phase at low undercoolings. As the melt undercooling prior to solidification increases theory predicts that the alloy undergoes a transition to solidification of a disordered FCC phase. The experimental results of this study for the solidification velocity as a function of undercooling indicate three regimes in the solidification behavior. First, at low undercoolings, the alloy solidifies in the ordered phase. Second, at intermediate undercoolings, the solidification is undergoes a transition from solidification of the ordered phase to solidification of the disordered phase. Third, at high undercoolings, the alloy solidifies as a disordered FCC phase.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 2/89 EXPIRATION: 8/94

PROJECT IDENTIFICATION: 962-25-08-20

NASA CONTRACT NO.: NAG8-765

RESPONSIBLE CENTER: MSFC

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**Presentations**

Bassler, B.T., Hofmeister, W.H., and Bayuzick, R.J. "Investigation of solidification velocity and microstructural genesis as functions of undercooling for gamma titanium aluminum." 3rd International Conference on High-Temperature Intermetallics, San Diego, CA, May 16-19, 1994.

Bassler, B.T., Hofmeister, W.H., and Bayuzick, R.J. "Ultra high speed imaging of electromagnetically levitated metals." 12th Symposium on Thermophysical Properties, Boulder, CO, June 19-24, 1994.

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*Studies of Nucleation and Growth of Intermetallic Compounds*

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PRINCIPAL INVESTIGATOR: Prof. Robert J. Bayuzick

Vanderbilt University

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CO-INVESTIGATORS:

W. Hofmeister

Vanderbilt University

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**TASK OBJECTIVE:**

The objective of this research is to quantitatively define the nucleation behavior of selected intermetallic compounds and the relationship between degree of undercooling and the speed of growth of the solid.

**TASK DESCRIPTION:**

This work focuses on the undercooling of intermetallic compounds and their pure metal constituents by containerless processing. This ground based research involves a continuation of already established drop tube work and electromagnetic levitation (on the bench). Specifics to be addressed are maximum undercoolings, nucleation frequency, the effect of undercooling on solidification velocity in refractory metals and alloys, and microstructural development as a function of deep undercooling/solidification velocity.

**Research Approach**

This work will consist of a comprehensive investigation on nucleation and growth of the solid from the undercooled liquid of selected intermetallics and their pure metal constituents. In order to do this, containerless processing is essential not only to eliminate the effect of the container on nucleation but also because the metals and alloys of interest are highly reactive and become contaminated with crucible material.

Three ground-based techniques for containerless processing will be applied to the effort. These are free fall in the long drop tube at the Marshall Space Flight Center, electromagnetic levitation on the bench at Vanderbilt University and electrostatic levitation on the bench at the Jet Propulsion Laboratory.

The intermetallic compounds chosen to be studied initially are TiAl and NiAl. In addition, studies will be done on pure Ti and pure Ni, as points of reference and because of their stand alone contribution to science. The particular intermetallics are chosen because both exhibit a wide solubility range as seen in their respective phase diagrams and, within compositions ranges, exhibit a tendency to be unencumbered by competition between alternate phase selection in the deep undercooling regime.

Nucleation frequencies as a function of temperature will be obtained for each combination of specimen type and ground-based processing technique. In each case, the nucleation probability distribution and the cumulative distribution will be constructed from the nucleation frequencies. To do this, a large number of undercooling experiments on each specimen type will be conducted by each of the ground based containerless methods. From analysis of the cumulative distributions, the kinetics of nucleation of the solid from the liquid for each specimen type under each processing condition will be quantitatively defined and examined in comparison to nucleation theory.

The ordered intermetallic alloys as well as their pure constituents will also be studied in order to determine the solidification velocity as a function of melt undercooling. The velocity will be determined using ultra high speed imaging to track the solidification front as it moves through the specimen. In opaque samples this can be done by monitoring the movement of the thermal field developed by recalescence. An instrument has been developed that consists of a 10 by 10 array of parallel tapped photodiodes and data acquisition. The system is capable of capturing data at rates of up to 1 million frames per second and the output from the array is calibrated for conversion to

temperature. For each alloy and pure element, a plot of the solidification velocity as a function of undercooling will be developed. This data will be compared to present solidification theories and any areas of discrepancy will be addressed. Of particular interest are the mechanisms controlling the rate of solidification at various undercoolings.

### TASK SIGNIFICANCE:

Intermetallic compounds and their composites are of considerable interest because of their potential use in high temperature structural applications. One strong driver for this interest is future goals for aircraft propulsion systems. Future aircraft engines will require high thrust-to-weight ratios and low specific fuel consumption, while remaining reliable, affordable and highly maintainable. Effort involving innovative designs, advanced materials and processing, and improved design and analysis tools is required to meet Integrated High Performance Turbine Engine Technology goals for doubling propulsion system capabilities by the year 2005. Benefits of the required increase in engine cycle temperature must not be offset by increased turbine weight or cooling air. Thus, new materials and design technologies are needed for advanced high-temperature turbines.

The materials of interest in this study show potential for use in such applications because of their significantly increased high temperature strength and acceptable low temperature damage tolerance. It is therefore necessary to fully understand the nucleation and solidification behavior of these intermetallic compounds.

### PROGRESS DURING FY 1994:

During the initial stage of this new grant the study of the solidification velocity as a function of undercooling for gamma titanium aluminum was continued. The previously gathered data was reexamined in order to improve the resolution of the measurements. Also, additional experiments were run for determination of the dependence of solidification velocity on undercooling.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-25-08-31

NASA CONTRACT No.: NAG8-1087

RESPONSIBLE CENTER: MSFC

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*Transport Phenomena During Equiaxed Solidification of Alloys*

PRINCIPAL INVESTIGATOR: Prof. Christoph Beckermann

University of Iowa

CO-INVESTIGATORS:

H.C. deGroh III

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

To investigate the macrosegregation and structural inhomogeneities resulting from gravity-induced thermosolutal convection and solid sedimentation during equiaxed solidification of alloys on a bulk level.

**TASK DESCRIPTION:**

The melt flow and solid transport phenomena occurring during equiaxed alloy solidification are investigated in experiments using both metal alloys and transparent analogues. The drag and heat/mass transfer coefficients of single equiaxed crystals are measured by employing fabricated dendrite models and, in situ using transparent model alloys. This information is combined to develop a multi-phase/-scale simulation model that allows for the calculation of the individual solid and liquid motions during solidification and incorporates the detailed phase interactions on a microscopic scale.

**TASK SIGNIFICANCE:**

This investigation complements NASA-sponsored research on the influence of fluid flow in alloy casting by including the important effects of gravity-induced motion of free equiaxed crystals. This combined experimental and theoretical/numerical study will provide (1) needed fundamental understanding of how liquid convection and the movement of free equiaxed grains interrelate and produce segregation and structural zones in castings, (2) progress towards a more complete numerical simulation model of transport and solidification phenomena, and (3) a base for defining future microgravity flight experiments on equiaxed dendritic solidification.

**PROGRESS DURING FY 1994:**

**1. Solidification Experiments**

The objectives of the bulk solidification experiments are to determine how convection in the liquid and settling/floating of free grains interrelate and result in segregation and structural patterns in castings, and to provide quantitative data for testing of the numerical model being developed. This will be achieved through a combination of metallic and transparent model (NH<sub>4</sub>C<sup>+</sup>-H<sub>2</sub>O) experiments.

The work on the metallic alloy experiments has concentrated on adapting and instrumenting the Glove Box Casting Facility and the Bulk Undercooling Furnace in the Microgravity Materials Science Laboratory at the Lewis Research Center. Experiments with various Pb-Sn alloys were performed, in which cooling rates, thermal gradients, and grains structure were measured. Modifications were made to achieve a more equiaxed structure. These experiments are presently being analyzed.

A complete set of transparent model alloy NH<sub>4</sub>C<sup>+</sup>-H<sub>2</sub>O experiments was conducted using a rectangular test cell. Temperatures were measured both along the walls (for later input into the numerical simulation model) and within the solution (for later comparison with the simulation results). Recalescence, a phenomenon typical of equiaxed solidification, was detected. The flow phenomena were recorded using time-exposure photography and shadowgraph visualization to detect density gradients within the liquid. Solidification was equiaxed, and extensive advection and sedimentation of solid dendrites were observed. These data are presently being compared to corresponding numerical simulations.

Additional solidification experiments were conducted using the transparent succinonitrile-ethanol system and the same test cell.

### 2. Measurements of Interfacial Transport Coefficients

A key feature of the proposed model is that the interactions between the solid and liquid phases are considered explicitly through the use of interfacial mass, momentum, energy, and solute balances. This, in turn, requires the knowledge of the drag coefficient and heat/mass transfer coefficients between the melt and the equiaxed crystals moving and growing in the undercooled melt. It is the objective of this part of the investigation to measure these coefficients. Generally, they are a complicated function of crystal microstructure, melt properties, relative velocity, and the number density of crystals present. The present experiments concentrate on single crystals, with the extension to multiparticle systems and packed beds of crystals accomplished through data available in the literature. Measurements are performed for plastic models of equiaxed dendrites and dendrite fragments, as well as for transparent model alloy crystals.

A correlation has been developed for the drag coefficient of single equiaxed crystals using measured settling velocity data for dendrite models fabricated from plastic. The correlation accounts for all relevant microstructural parameters, including the overall width of the crystal, the crystal envelope sphericity, the interdendritic arm spacings, and the internal solid fraction -- all parameters used in the numerical simulation model. This also allows for the application of the correlation to real metallic systems and transparent model alloys, and good agreement was achieved with our measurements in  $\text{NH}_4\text{C}^+-\text{H}_2\text{O}$ .

The same correlation framework was then extended to multiparticle systems, unifying the above single crystal drag data with permeability measurements on packed beds of equiaxed crystals available in the literature. The correlation also provides information on the partitioning between the flows around and through the dendrites before packing. The correlation has been implemented in the numerical simulation model.

Measurements of the heat/mass transfer coefficients of single growing and settling equiaxed transparent model alloy crystals are presently underway. A settling column has been constructed that allows for the precise control of the undercooling and the introduction of equiaxed crystals. Measurements of the growth rate and settling velocity will be used to calculate the heat/mass transfer coefficient. Both  $\text{NH}_4\text{C}^+-\text{H}_2\text{O}$  and succinonitrile-ethanol solutions will be used.

### 3. Model Studies

A two-phase model of alloy solidification has been extended to include equiaxed dendrites by introducing a new multi-phase/-scale concept. The model was first tested for the limiting case of diffusion controlled solidification, as can be expected in microgravity. The predictions were compared with experimental data available in the literature for purely columnar, purely equiaxed, and coupled columnar/equiaxed growth. In particular, the coupled columnar/equiaxed experiments corresponded only approximately to a convection-free situation, making a true validation of the diffusion model impossible. It was concluded that, given a realistic nucleation model, the columnar-to-equiaxed transition can be predicted for a variety of alloys, compositions, and cooling conditions. Final validation of the model, however, must await experiments in microgravity.

Numerical simulations that include melt convection and solid transport have been carried out using the two-phase model. The primary purpose of these exploratory simulations was to examine the influence of the equiaxed nuclei density on the solid transport behavior and to compare with results for a fully stationary solid phase. The simulations were performed for solidification of an Al-4wt%Cu alloy in a rectangular test cell cooled from the left sidewall. Three simulations were performed: (a) stationary solid, (b) an average equiaxed nuclei density of  $10^{11} \text{ 1/m}^3$  and (c) of  $10^9 \text{ 1/m}^3$ . The mushy zone structure was substantially different for all cases. In cases (b) and (c), a sediment layer of equiaxed crystals formed at the bottom in a manner similar to that experimentally observed. Final macrosegregation patterns and grain size distributions were also predicted and compared. Presently, quantitative comparisons between the  $\text{NH}_4\text{C}^+-\text{H}_2\text{O}$  experiments and corresponding simulations are being attempted.



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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	2	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

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TASK INITIATION: 11/93    EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-25-05-23

RESPONSIBLE CENTER: LeRC

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### *Gravitational Effects on the Development of Weld-Pool and Solidification Microstructures in Metal Alloy Single Crystals*

PRINCIPAL INVESTIGATOR: Dr. Lynn A. Boatner

Oak Ridge National Laboratory

#### CO-INVESTIGATORS:

S. David

Oak Ridge National Laboratory

G. Workman

University of Alabama, Huntsville (UAH)

#### TASK OBJECTIVE:

The objectives of this research are to achieve an in-depth quantitative understanding of the role played by convection-driven and surface-tension-driven heat and mass transport in determining the shape and surface morphology of fusion weld pools and, thereby, to control the nature of the solidification processes that determine the microstructural and mechanical properties of welds.

#### TASK DESCRIPTION:

By combining ground-based results with those obtained in the low- and high-g environments provided by NASA aircraft, detailed microstructural information can be obtained through the use of single-crystal alloy specimens, and both gravitational and surface-tension-driven effects on weld-pool formation and microstructural properties can be delineated and quantified. The results of these investigations are currently yielding new information that is serving to advance our level of understanding of various phenomena which determine both weld-pool shapes, mass transport, and the morphological properties of solidification surfaces.

This research represents a new and innovative approach to the investigation of gravitational effects on melt-pool shapes, solidification phenomena, and weld microstructures. This approach is based on the application of our recently developed single-crystal methods for the delineation of solidification microstructural properties and on the new quantitative analytical methods that have been developed in conjunction with the unique experimental results that can be obtained through the application of these single-crystal techniques. This approach to the study of weld and solidification microstructures begins with the growth of macroscopic single crystals of the alloy system that is to be investigated. As in the case of our previous ground-based studies, Czochralski-grown single crystals of the pure ternary alloy 70Fe-15Ni-15Cr (a compositional analog of one of the 300 series of stainless steels) are being utilized.

#### TASK SIGNIFICANCE:

The long-term goal of this research effort is to achieve a firm scientific basis for the development of a comprehensive scientific program in which more-complex solidification and welding experiments can be carried out on NASA aircraft, on Space Shuttle flights, and eventually on Space Station Freedom. Since the microstructural properties of weld pools and the distribution of certain impurities within the weld are central to determining the weld mechanical strength, these experiments have practical implications regarding a wide range of construction, fabrication, and manufacturing operations both in space and on earth.

#### PROGRESS DURING FY 1994:

A comprehensive set of solidification experiments has been completed in which single crystals of the ternary alloy 70Fe-15Ni-15Cr were utilized in investigations of microstructural formation in stationary melt pools formed by both electric-arc and electron-beam melting. This alloy is a pure analog of a stainless steel in the 300 series of commercial stainless alloys. By carrying out solidification experiments in which stationary melt pools were formed on stainless steel single-crystal surfaces that were oriented to coincide with (100), (110), and (111) principal

crystallographic planes, it was possible to carry out a three-dimensional reconstruction of the dendritic-solidification microstructures corresponding to melt pools with various shapes. This three-dimensional reconstruction was achieved by using information provided by optical-metallographs which were obtained by sectioning the solidification microstructures along various principal crystallographic directions.

Most significantly, in the course of this research activity, a unique surface morphology that is present following the solidification of stationary melt pools formed by either electric-arc or electron-beam melting was observed. This surface morphology consists, in general, of concentric surface undulations, or "ripples." These undulations begin at the outer circumference of the melt pools and continue in toward the center with a steadily decreasing diameter.

Experiments using alloy single-crystal specimens have been initiated for the purpose of studying mass-transport effects in both low- and high-g environments for stationary melt pools formed in a high-temperature alloy system such as stainless steel. These investigations have utilized the controlled introduction of a "tracer" element into the bottom of the melt pool subsequent to its formation. The solidified melt pool is then cross sectioned and back-scattered-electron elemental analysis is used to map out the distribution of the tracer element in the solidified melt pool. Although these studies were initially conceived as an independent investigation of mass-transport phenomena in low- and high-g conditions, subsequent to the investigations of surface undulations noted above, it was determined that, in fact, the nature of mass transport in these systems and under these conditions is apparently not independent of the effects leading to the formation of surface ripples on solidified stationary melt pools. Accordingly, the tracer technique, which has been developed in the course of this work, is now being applied to a combined investigation that includes an in-depth study of the formation of surface undulations and mass transport in stationary melt pools formed by electric-arc, electron-beam, and laser melting.

A new laser-melting system has been assembled by co-investigators Gary Workman and Guy Smith at the University of Alabama - Huntsville, and this new, more-powerful system will be used in a series of melting and solidification experiments to be performed in the latter part of October, 1994 on the KC-135 aircraft. The melt-pool shapes and associated microstructural properties and the surface morphologies formed during the low- and high-g portions of the KC-135 flight path will be investigated in the course of these experiments.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	0	PhD Degrees:	0

**TASK INITIATION:** 2/93 **EXPIRATION:** 2/96**PROJECT IDENTIFICATION:** 962-25-08-23**NASA CONTRACT NO.:** H-13059D**RESPONSIBLE CENTER:** MSFC

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### *Modelling of Convection and Crystal Growth in Directional Solidification of Semiconductor and Oxide Crystals*

PRINCIPAL INVESTIGATOR: Prof. Robert A. Brown

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

D. Bornside

Massachusetts Institute of Technology (MIT)

#### TASK OBJECTIVE:

The research effort focuses on the development of a detailed understanding of the interactions of heat, mass and solute transport on the quality of crystals grown from melt by the vertical Bridgman (VB) method. Both semiconducting alloy (GeSi) and oxide (Bismuth germanate) crystals are being studied.

#### TASK DESCRIPTION:

The research is aimed at developing a detailed simulation for VB crystal growth systems for semiconductor and oxide materials that begins to relate macroscopic analysis of heat, mass, and solute transport with the microscopic properties of the grown crystal. The analysis and simulation tools developed will be applied to the analysis of several crystal growth systems. The binary alloy GeSi and the pseudo-binary BGO Bismuth germanate are selected for analysis of VB growth. The research is divided into three sections:

- Development of integrated model for vertical Bridgman growth,
- Parallel processing and simulation of three-dimensional convection,
- Application to GeSi alloy crystal growth, and BGO crystal growth.

Fundamental understanding of the interactions of heat, mass, and solute transport on the quality of crystals grown from the melt is important in the design and control of systems for crystal growth in microgravity and for the interpretation of the results of experiments performed both on Earth and in space. The research program focuses on the development of the detailed analysis of these features in the vertical Bridgman (VB) crystal growth of semiconductor crystals and oxide materials used in optoelectronic applications. The analysis has two parallel goals: to develop the first integrated model and numerical analysis for the growth of these materials that accounts for the details of the design and operation of the furnace; and to link the predictions of the macroscale analysis of heat transfer and convection with the quality of the crystal as measured by the number of crystallographic defects and the compositional homogeneity of the material. The research integrates several aspects of research that are ongoing at M.I.T. to accomplish these goals: the development of numerical analysis for integrated heat transfer throughout a high temperature furnace, including internal radiation in a semitransparent material; the modeling of dislocation motion and multiplication in semiconductor materials; and applications of new robust algorithms for parallel computation.

The analysis of coupled furnace design and the prediction of material quality will be applied to two distinct crystal growth technologies that have potential application for crystal growth in microgravity: the growth of GeSi semiconductor alloys, a substrate material used for superlattices, and the growth of Bismuth Germanate or BGO, a scintillating oxide material used in high energy detector applications.

#### TASK SIGNIFICANCE:

The techniques for analysis and the quantitative insights developed in this research have broad application to a variety of ground-based and space flight experiments supported by NASA and its European counterpart, ESA. Many of the previous research results conducted in this research area represent theoretical collaboration with experiments supported by NASA. These interactions between detailed modeling and experiments are crucial to the

design of well-controlled experiments for space, as well as optimized crystal growth on Earth. The development of a user-friendly simulator for VB growth systems will be a direct result of this program and will be made available to others in the research community.

#### PROGRESS DURING FY 1994:

The research program described here focuses on the development of the computational tools for detailed analysis of the vertical Bridgman crystal growth system for the growth of both semiconductor and oxide crystals. The research links several components to accomplish these goals. The first is the development of very accurate and efficient methods for the computation of diffuse-gray radiation in the furnace enclosure and of radiation in a semitransparent material for the case of oxide growth. The second is the development of advanced computational methods capable of computing three-dimensional convection in the melt coupled with realistic furnace models for analysis of melt convection during growth under terrestrial and microgravity conditions. These algorithms will be based on the application of finite element methods for the solution of the integrated heat transport and convection models for vertical Bridgman growth using MIMD parallel computers.

Research has been directed toward the development of computational tools needed for the vertical Bridgman simulator described above. To date the following developments have been made:

1. A new object-oriented method for the computation of view factors for axisymmetric, diffuse-gray radiation has been developed.
2. The basic elements of a parallel implementation of the finite-element/Newton method for solving transport problems has been completed and demonstrated for buoyancy-driven convection.

#### Object-Oriented View Factor Calculation

Accurate calculation of view factors for use in the diffuse-gray radiation calculation is one of the most important elements of calculation of high temperature radiation in crystal growth systems. Two features of the numerical method are needed to insure accuracy of the radiation calculation: fine finite element surface discretizations and accurate calculation of the view factor from one surface element to another. Most previous calculations of crystal growth furnaces use some form of the Facet algorithm first modified by us for this application. Here the view factor from any flat surface element to any other is computed by a double area quadrature, once the possibility of blocking by another surface element is tested for each element. The computation of the blocking is the most time intensive part of the algorithm, scaling as  $O(N_s^3)$ , where  $N_s$  is the number of surface elements in the radiation enclosure.

We have developed an object-oriented method (OOM) that takes account of the axisymmetry of typical furnace geometries and that groups surface elements into conical and cylindrical objects, thereby drastically reducing the blocking calculation. The OOM is based on Nusselt's projection method for computing view factors by projection on the surface of a hemisphere. The OOM method scales as  $O(N_{obj}N_s)$ , where  $N_{obj}$  is the number of objects in the enclosure; usually  $N_{obj} \ll N_s$ . Simulations for model systems and for realistic crystal growth furnaces have verified this scaling.

#### Parallel Implementation of the Finite-Element/Newton Method

Robust numerical solution of very detailed simulations of crystal growth furnaces and three-dimensional convection in the melt present computational problems that are at the leading edge of supercomputing today. To advance these simulations will require the application of parallel computing to the solution of the physiochemically complex transport models that are needed to model crystal growth systems. A major goal of this program is the development of a finite-element based methodology for this purpose. Our approach is to use finite element methods to reduce these models to large systems of algebraic equations and to solve these equations by Newton's method. Each Newton iteration requires the solution of a large set of algebraic equations which is the heart of the algorithm. We do this by LU decomposition using domain decomposition and nested-dissection to distribute the work of LU

decomposition over the multiple processors of a MIMD parallel computer. Our implementation of LU decomposition is among the fastest algorithms of its type available. We have used this algorithm to solve model problems in steady-state and transient buoyancy-driven flow and have demonstrated the robustness of the algorithm, as well as the linear speed increase of the algorithm with increasing number of processors.

#### Dynamics of Small-Scale Floating Zones

Small-scale floating zones are used in a number of microgravity experiments and offer a very interesting case study in the dynamics of meniscus-defined crystal growth. The goal of this project has been to develop detailed models and numerical simulation tools for the analysis of steady-states, their stability, and nonlinear dynamics of small-scale floating zones. The thermocapillary models include both steady-state and transient analysis of heat transfer in all phases, convection in the melt, the shapes of the melt/crystal and melt/feed-rod interfaces and the shape of the meniscus. Finite-element analysis is used to discretize the model and Newton's method is used to solve the nonlinear algebraic equation set that arises in the steady-state calculation and at each time-step of a fully implicit time integration. Zone stability is tested by linear stability theory implemented in the finite-element formalism using an Arnoldi algorithm to compute the most dangerous eigenvalues of the linear stability problem for each set of operating conditions. Our initial results describe the dynamics of the zone in the absence of convection in the melt. Here the interplay between surface tension, which limits the zone length through the Rayleigh limit, and heat transfer are shown to lead to a new oscillatory mode for instability in the floating zone shape in which the zone length oscillates with growing amplitude until the zone breaks. This instability occurs before the maximum zone length that is predicted by the Rayleigh limit for an ideal cylindrical zone. Closed-form linear and nonlinear analysis demonstrate that the zone dynamics associated with the Rayleigh limit for growing floating zones is altered from the conventional dynamics seen for captive cylindrical drops used as model systems.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 7/93 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-21-08-17

NASA CONTRACT NO.: NAG8-961

RESPONSIBLE CENTER: MSFC

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*Microstructure Formation During Directional Solidification of Binary Alloys Without Convection: Experiment and Computation*

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PRINCIPAL INVESTIGATOR: Prof. Robert A. Brown

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

A combination of experimental and theoretical research will be aimed at developing a predictive understanding of cellular and dendritic microstructures of dilute binary alloys and lamella spacing in the growth of eutectics formed during thin-film solidification. The results will form the basis for prediction of the dependence of microstructure formation on macroscale properties of bulk solidification systems, such as the imposed temperature gradient and the sample growth rate.

**TASK DESCRIPTION:**

The extensive theoretical and experimental research on microstructure formation point to the formation of cellular microstructures in binary alloys and lamella eutectics as being formed under conditions of long time-scale, spatiotemporal chaos in the pattern. These patterns involve a band of wavelengths that evolve with changes in the operating conditions, such as growth rate and temperature gradient, and include very long wavelength interactions through which the pattern communicates over length scales much larger than the characteristic cell size. The outstanding problem that will be addressed in the research is to begin to construct mean field models for solidification microstructure that are based on microscopic mechanics of individual elements in the microstructure. Experimental, theoretical and computational studies will be conducted that will lead toward this goal. These elements of the research plan are:

- Experimental studies of spatiotemporal chaos in cellular solidification and the role of externally applied forcing functions on regularization of the pattern;
- Extension of the analysis of wavelength selection to thin-film eutectic solidification;
- Experimental studies of lamellar eutectic growth in thin-film solidification; and
- The development of stochastic and mean field models for pattern formation in directional solidification.

The microstructure of metal alloys formed from directional solidification plays an important role in the mechanical and electrical properties of these materials. This investigation describes experimental and theoretical research aimed at developing a predictive understanding of cellular and dendritic microstructures of dilute binary alloys and the lamella spacing in the growth of eutectics formed during thin-film solidification. The combination of experimental and theoretical results will form the basis for prediction of the dependence of microstructure formation on macroscale properties of bulk solidification systems, such as the imposed temperature gradient and the sample growth rate. The experimental studies are carefully designed so that bulk convection, driven by density gradients, is unimportant. In this way, the results are applicable to gravity-free experiments that will be undertaken during space flight. The theoretical framework for understanding nonlinear pattern formation during solidification is so complex that there is little hope of unraveling the mechanisms for pattern formation in the presence of convection without rigorous analysis and experiment in the absence of convection. The thin-film experimental geometry offers the only mechanism for accomplishing this goal on Earth.

**TASK SIGNIFICANCE:**

The research has the promise of making significant progress towards the development of a theoretical framework for characterizing the formation of microstructure in alloy solidification. The experiments and microscale calculations

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to be conducted will lay the foundation for detailed description of the mechanisms for length scale adjustment in cellular and lamellar eutectic growth. The development of kinetic theory for describing the evolution of the microscale will give the connection between processing variables and microscale formation that is necessary for adaptation of the theory to practice in industrially important systems, and will provide a framework for the characterization of alloy systems for microstructural modeling parameters.

#### PROGRESS DURING FY 1994:

Research in FY94 focussed on two fronts: refinement of experimental methods for the measurement of the dynamics of large collections of cells during solidification of a binary alloy and the development of simple models that exhibit dynamics similar to what is observed in the experiments.

#### Experiments Using Thin-Film Solidification System

During FY94 we have refined the experimental methods for solidification of succinonitrile-acetone samples in a large-scale thin-film directional solidification system developed by us (C.T. Lee and R.A. Brown, *Physical Review B* 47 4937 (1993)). This system is unique because it allows for long-time (approximately 5000 diffusion time scales) solidification of well-controlled samples. Experiments this year have focussed on the development of Fourier Transform Infrared Spectroscopy (FTIR) as an analytical measurement of the composition of dilute acetone in succinonitrile samples and the use of the system for precise measurement of the conditions for the onset of morphological instability in the thin-film solidification system. Data from these experiments have been compared with the constitutional supercooling criterion and the stability boundary from the complete linear stability analysis for the onset of morphological instability. The comparison is excellent for the critical value of the growth rate ( $V_{crit}$ ) for a given alloy concentration ( $c_0$ ) and temperature gradient ( $G$ ). However, the structure of the cells seen in the experiments neither resembles the sinusoidal cells predicted by the linear stability analysis nor has a dominant component of the length scale of approximately 500  $\mu\text{m}$  predicted by the linear analysis to be most dangerous. Instead, the length scales of the cells are approximately 100  $\mu\text{m}$  and the cells are irregular. We are in the process of developing a new interfacial imaging system for rapid statistical processing of the cellular structures for quantitative determination of the interfacial morphology.

#### Modelling of Nonlinear Dynamics of Cellular Solidification

Nonlinear simulations of large collections of cells for solidification over long time scales are exceedingly expensive and make employing any of the tools for calibrating the degree of spatiotemporal chaos in the pattern difficult to employ. Accordingly, we have begun to search for simple nonlinear amplitude equations which give the same qualitative nonlinear dynamics as the solutions from the full model that results from solving continuum transport models for solute and heat transfer coupled with the Gibbs-Thomson condition for interfacial equilibrium. When the temperature field is held fixed, this description is known as the Solutal Model. Several features of the nonlinear dynamics in the Solutal Model need to be captured by the amplitude equation:

- (1) The neutral stability curve must be very flat so that a range of spatial wavelengths go unstable at values of the dimensionless growth rate (the control parameter) that are only slightly above the most critical value.
- (2) The steady-state nonlinear solutions of the equation must demonstrate the secondary bifurcating pattern seen in the Solutal Model and which is responsible for the rapid decrease in the wavelength from the critical value for small increases in growth rate.
- (3) The dynamics of large collections of cells should demonstrate the dynamics involving the tip splitting of cells, which leads to the birth of new cells and the death of cells between the grooves of others.

We have developed a nonlinear evolution equation that demonstrates these features. It combines a non local linear operator - expressed as an integral operator in real space - with a quadratic nonlinearity similar in form to the Kuramoto-Sivashinsky equation. We are in the process of studying the weakly and strongly nonlinear dynamics of this class of equations using both asymptotic and numerical methods.



Effect of Convection on Evolution of Cellular Morphologies

The role of convection in effecting the evolution of cellular morphologies from the onset of morphological was investigated by two-dimensional numerical simulations in which thermosolutal convection was added to the Solutal Model described above. The most important finding from these calculations was the difficulty associated with determining the difference between the so-called morphological and convective modes for instability for finite amplitude cells. We have demonstrated that nonlinear states created by both types of instabilities lead to cells separated by very deep grooves for conditions only slightly in excess of those for neutral stability. This work is being prepared for publication.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 7/93 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-25-08-25

NASA CONTRACT No.: NAG8-962

RESPONSIBLE CENTER: MSFC

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*Evolution of Crystal and Amorphous Phase Structure During Processing of Thermoplastic Polymers*

PRINCIPAL INVESTIGATOR: Dr. Peggy Cebe

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

Our objective is to study microstructure evolution during melt processing and secondary thermal treatment of high performance thermoplastic polymers which are candidate materials for advanced composite applications. We will investigate formation of structure in both the crystalline and the amorphous phases. The purpose of the study is to determine the effects of processing conditions on the resultant structure and physical properties of thermoplastics. We hope to address the manner in which self deformation stresses affect the microstructure of the solidifying polymer melt. Processing in a microgravity environment will impose very different stress states on molten polymers during solidification, compared to ground based processing conditions. Dependence of polymer microstructure and physical properties on processing conditions can more easily be predicted and controlled under microgravity conditions where self deformation forces are avoided.

**TASK DESCRIPTION:**

Present ground based processing methodologies requires gravity compensation to support the processed piece during solidification. Therefore nearly all melt processing approaches (eg., extrusion, film blowing, fiber drawing) involve very rapid quenching of the molten material to avoid imposing self deformation stresses on the melt. Rapid quenching results in inhomogeneous cross-sections, since the center of the piece cools more slowly than the exterior. In a microgravity environment, it would be possible to use very slow melt processing techniques to obtain more uniform microstructure throughout the section, minimizing the morphology gradient between the skin and the core of the section.

The proposed research is a ground based study of slow melt processing in the gravity environment. We will process thermoplastic polymers by solidification from the melt without gravity compensation. First we will use real-time x-ray scattering at elevated temperature to study the kinetics of solidification. Then we will examine the resultant microstructure at room temperature after processing. Self deformation stresses on the melt will be studied to determine the effects of gravity on the development of crystalline structure and morphology from the (stressed) amorphous melt. We will characterize the unit cell structure of the crystal phase, the disposition of unit cells within the crystals, and the organization of crystals into larger units such as spherulites, fibrillar bundles, or shish-kebab structures. We will also study the amorphous phase structure to determine degree of chain alignment and the amount of rigid vs. mobile amorphous phase. Additionally, the location of rigid vs. mobile amorphous chains with respect to the crystals will be examined.

The principal approaches used in this study will be real-time wide and small angle x-ray scattering, which will be performed at the Brookhaven National Synchrotron Light Source and in our in-house x-ray laboratory. In addition, we will use differential scanning calorimetry, optical microscopy and birefringence, optical waveguide prism coupling, scanning electron microscopy, and dynamic mechanical spectroscopy to characterize the polymers after processing. Standard mechanical tests, such as stress-strain and impact resistance measurements, will be used to study the performance of the test pieces. One unique aspect of the proposed research is the combined study of both the crystal and the amorphous phases. We will be concerned not only with the nucleation and growth of crystals, but also with residual stresses and chain alignment in the amorphous phase.

**TASK SIGNIFICANCE:**

Our ultimate goal is to contribute to a fundamental understanding of microstructure evolution in thermoplastic polymers solidifying under influence of self deformation stresses, and to determine the ameliorating effects of the microgravity environment. Once we know the effect of self deformation stress during processing, we will be better able to predict the solidification behavior of polymers in the microgravity environment. This will lead to development of unique processing applications that capitalize on the microgravity environment. Complementary experimentation under reduced gravity conditions would later be proposed to be performed on the Space Shuttle, or on Space Station. Ground based facilities, such as drop towers and KC-135 would not provide the capability for real-time studies of microstructure development under practical processing conditions.

**PROGRESS DURING FY 1994:**

We are awaiting the start of this project in late 1994, or early 1995.

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**STUDENTS FUNDED UNDER RESEARCH:****TASK INITIATION: 12/94    EXPIRATION: 12/96****PROJECT IDENTIFICATION: 962-26-08-12****NASA CONTRACT No.:NAG8-1110****RESPONSIBLE CENTER: MSFC**

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*Optical Properties for High Temperature Materials Research*

PRINCIPAL INVESTIGATOR: Dr. Ared Cezairliyan

National Institute of Standards and Technology (NIST)

CO-INVESTIGATORS:

S. Krishnan

Containerless Research, Inc.

J. McClure

National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE:**

The objective of this research is to obtain definitive values for the normal spectral emissivity of selected high-melting-point metals by two independent techniques in order to provide a foundation for reliable radiometric temperature measurements in materials research at high temperatures, both in microgravity and on the ground.

**TASK DESCRIPTION:**

The research will include accurate measurements of the normal spectral emissivity of selected metals near and at their melting points in a series of subsecond pulse-heating experiments in which the emissivity will be determined at two laser wavelengths in the range 0.5-0.9  $\mu\text{m}$  by two independent techniques involving high-speed pyrometry and laser polarimetry, respectively. The simultaneous measurements by the two techniques on the same specimen will minimize a number of major experimental uncertainties, in particular those arising from specimen surface conditions and specimen purity.

**TASK SIGNIFICANCE:**

The results of this research will enable the establishment of reference values for normal spectral emissivity (also leading to high temperature radiometric standards) which are critically needed for accurate temperature measurements in materials research on high-temperature liquids and melts and in the determination of their thermophysical properties at high temperatures, under either microgravity or terrestrial conditions. In addition, the proposed work will resolve the current major controversy in the scientific literature regarding the wavelength dependence of normal spectral emissivity of metals at and near their melting points.

**PROGRESS DURING FY 1994:**

The laser polarimeter developed by the Containerless Research Inc. was integrated into the millisecond-resolution pulse-heating system at the National Institute of Standards and Technology. The required modifications in the polarimeter and the pulse-heating system were made. A new digital data acquisition system was added to the system and computer programs were prepared for processing of data. Combined operation of the entire system, including the polarimeter and pyrometer instrumentation was tested. Preliminary measurements of the normal spectral emissivity in the red region (600-650 nm) have been performed on selected metals in their solid phase (Mo, W) and at their melting temperatures (Ni, Zr). For the tubular Mo specimen, the agreement between the emissivities obtained by the two independent methods (pyrometric and polarimetric) in the temperature range 1600-2400 K was 2-3%. Experiments on a solid cylindrical W specimen in the range 1700-3000 K yielded emissivity values from polarimetric data in agreement, within 2%, with data reported in the literature. Experiments on Ni and Zr strips at their melting temperatures yielded emissivities which show a reasonable agreement (2-4%) between the results obtained by the two techniques.

The preliminary experiments suggested improvements in the system. The improvements in the pyrometer and polarimeter have already been completed; those in the experiment chamber and specimen preparation are underway. After the completion of all the improvements, definitive accurate measurements of the normal spectral emissivity in the red region will be performed on selected refractory metals (such as Mo, W) as well as industrially important metals (such as Fe, Ni, Ti). Similar measurements will also be performed in the near infrared region (around 900

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

nm). The results of successful experiments will assure the reliability of direct measurements of normal spectral emissivity with the laser polarimetric technique and will provide much-needed accurate data on the emissivity of selected high temperature materials.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 3/93 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-25-08-27

NASA CONTRACT No.: H-18067D

RESPONSIBLE CENTER: MSFC

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Cezairliyan, A., and Krishnan, S. "Optical properties for high temperature materials research." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

Cezairliyan, A., Krishnan, S., and McClure, J.L. "Laser polarimetric and spectral radiometric measurements of the normal spectral emissivity of high temperature solids in pulse-heating experiments." 12th Symposium on Thermophysical Properties, Boulder, CO, June 21, 1994.

*Fluid Mechanics of Directional Solidification at Reduced Gravity*

PRINCIPAL INVESTIGATOR: Prof. Chuan F. Chen

University of Arizona

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

## TASK OBJECTIVE:

The primary objective of this research is to provide ground-based support for the Casting and Solidification Technology (CAST) flight experiment, flown on IML-1 in January 1992. The secondary objective of the proposed research is to examine the stability phenomena associated with the onset of freckles and the mechanisms for their subsequent growth and decline (to the eventual demise of some).

## TASK DESCRIPTION:

The focus is to study the convective motion and freckle formation during directional solidification of  $\text{NH}_4\text{Cl}$  from aqueous solution at simulated parameter ranges equivalent to reduced gravity. This will involve state-of-the-art imaging techniques and mathematical models for the prediction of the observed phenomena.

## TASK SIGNIFICANCE:

Once the instability phenomenon that causes the onset of freckles in the casting of alloys is understood, then we can devise means to manufacture castings free of defects, both in space and on earth.

## PROGRESS DURING FY 1994:

We have successfully carried out an experiment using x-ray tomography (CAT Scan) to determine the solid fraction distribution in a growing mushy layer in a  $\text{NH}_4\text{Cl}$  solution being directionally solidified from below. The results show good agreement with the prediction of Amberg and Homsy (Journal of Fluid Mechanics, 1993). The results also show, contrary to intuition, there is a significant decrease of solid fraction towards the bottom of the tank where the temperature is the lowest. This occurs at the later stages of the solidification process when the chimneys are fully developed. As a result, the increase in local permeability can be as much as 50% thus providing a passage for the fluid supplying the plumes to flow along the bottom of the mushy layer.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 5

TASK INITIATION: 4/91 EXPIRATION: 3/94

PROJECT IDENTIFICATION: 962-25-05-21

NASA CONTRACT NO.: NAG31268

RESPONSIBLE CENTER: LeRC

## BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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*Microgravity Chemical Vapor Deposition*

PRINCIPAL INVESTIGATOR: Dr. Ivan O. Clark

NASA Langley Research Center (LaRC)

## CO-INVESTIGATORS:

W.A. Jesser

University of Virginia

P.V. Hyer

Lockheed Engineering &amp; Sciences Co.

E.J. Johnson

Lockheed Engineering &amp; Sciences Co.

**TASK OBJECTIVE:**

This research will develop a better understanding of the scientific principles underlying chemical vapor deposition (CVD). The proposed research will determine to what extent microgravity can elucidate and separate these competing phenomena and will form the basis for a proposal to perform a series of flight experiments to more fully elucidate these scientific principles.

**TASK DESCRIPTION:**

Ground-based experimental and numerical investigations will provide both basic scientific information on the heat and mass transfer effects central to the CVD process and define specific follow-on reduced-gravity investigations. This multi-pronged approach will maximize the utilization of available resources and capabilities. In the numerical modeling, both finite difference and spectral element techniques will be used and the predictions compared. In the experimental phases of the effort, a horizontal CVD reactor design will be used for the growth of a model material, such as aluminum, and a commercially important material, InP. Laser velocimetry measurements of the flow fields in the reactor will also be performed.

**TASK SIGNIFICANCE:**

CVD is an extremely important industrial technique with applications in the fields of semiconductors, optics, and corrosion resistance. The nature and quality of the layers formed are dependent on mass and energy transport as well as homogeneous and heterogeneous chemical reactions and nucleation. Commercial CVD processes currently employ reactors developed through decades of empirical trial and error. Scientific understanding of the CVD process is limited by the difficulty of separating the heat and mass transport due to externally forced convection and that of the internal processes of buoyant thermal convection, buoyant solutal convection, and thermal (Soret) and solutal diffusion. There is also forced convection due to volume changes arising from both reactive chemistry and thermal effects. A better understanding of these effects is essential to achieve desired improvements in perfection, uniformity, and size of grown layers and to provide an engineering design basis for these systems.

**PROGRESS DURING FY 1994:**

The design of the test vessel is being refined using a parametric numerical study of geometric vs analytical resolution requirements for measuring the reactor effects. A candidate organometallic material has been selected and efforts are underway to develop the thermophysical properties database necessary to accurately model deposition.

Thermal imaging techniques have been applied to a model chemical vapor deposition (CVD) reactor. The thermal imaging provided much improved boundary conditions for use in the numerical modeling effort. It also demonstrated that jets were present in the reactor when hydrogen test gas was used and that the thermal image of these jets could be used as a diagnostic tool to improve the repeatability of experiments in CVD reactors.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-21-06-09

RESPONSIBLE CENTER: LaRC

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**Presentations**

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*Glass Formation and Nucleation in Microgravity: Containerless-Processed, Inviscid Silicate/Oxide Melts (Ground-Based Studies)*

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PRINCIPAL INVESTIGATOR: Dr. Reid F. Cooper

University of Wisconsin, Madison

## CO-INVESTIGATORS:

J.H. Perepezko

University of Wisconsin, Madison

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TASK OBJECTIVE:*1. Nucleation by internal oxidation or reduction of transition metal-bearing silicate glasses and melts.*

If a change in valence state of a transition metal cation within a silicate melt is associated with a change in its structural role within the melt, one might be able to effect internal homogeneous nucleation within the melt via a change in the external environment, for example, by a redox reaction. Critical to the hypothesis is the nature of transition metal cations to make the melt into a semiconductor: Conduction electrons or electron holes are majority defect species and thus serve to decouple cation and anion diffusion fluxes that occur in an oxygen chemical potential gradient. One consequence is that oxidation or reduction reactions can occur internally (i.e., within the body of the melt) instead of solely on the surface. These reactions can result in the destabilization of the melt such that crystallization reactions occur in finely (nm-scale) dispersed regions of the melt body (e.g., the formation of  $\text{Fe}^{3+}$ -bearing spinel precipitates via the internal oxidation of an originally  $\text{Fe}^{2+}$ -bearing aluminosilicate melt). One can thus create fine-grained glass-ceramics from what would normally be non-glass-forming melts. Specific research involves control reaction experiments on silicate glasses and levitated reaction experiments (aero-acoustic and electrostatic levitation) on silicate melts.

*2. Internal nucleation of inviscid pseudobinary silicate melts via metastable liquid-phase immiscibility.*

Binary alkaline Earth oxide-silicate melts are highly exothermic. Nevertheless, the structural variations between highly polymerized (silica-rich) and poorly polymerized (silica-poor) silicate liquids result in the creation of composition zones (on the silica-rich end of the phase diagram) where a single silicate liquid is not stable. On the silica-poor end of the diagram, this immiscibility would be metastable. As a consequence, if one can sufficiently undercool an inviscid, silica-poor melt, one could perhaps cause metastable amorphous-phase separation to occur prior to any crystallization. The phase separation could further promote the internal, fine ( $\mu\text{m}$ )-scale, uniform nucleation and crystallization of the material. The creation of unique glass-ceramic materials becomes a possibility. Scientifically, measurements of heat evolution rate in such droplets will address questions concerning the role of amorphous phase separation in crystalline nucleation.

## TASK DESCRIPTION:

Two research approaches are employed for the two tasks:

1. Containerless processing for oxidation of  $\text{Fe}^{2+}$ -bearing alkaline Earth aluminosilicate melts via aero-acoustic levitation (AAL). Small droplets (~3mm diameter) of ferrous iron-bearing calcium aluminosilicate glass, prepared initially by bulk melting in a controlled-oxygen activity furnace, are levitated and remelted using AAL and laser heating. The droplets thus formed are evaluated for their surface reactions, using Rutherford backscattering spectroscopy (RBS), and for their internal reactions using analytical transmission electron microscopy (AEM and TEM). The kinetics of the redox reaction are evaluated as functions of temperature, time, and oxygen activity, the latter controlled via the gas used as a levitation medium. The results of these experiments are compared to those done at low temperature on glasses of identical composition; with such a check, the study can be later extended to melts too inviscid to be glass formers. The nature of nucleation as affected by local oxygen fugacity will be explored using electron diffraction study of the internal oxidation front.

2. Drop-tube processing of pseudobinary silicate melts. Binary  $\text{MgO-SiO}_2$  metasilicate compositions near the deep cristobalite ( $\text{SiO}_2$ )-enstatite ( $\text{MgSiO}_3$ ) eutectic are melted in a drop tube. Initially fine, crystalline powder, the fine

droplets are allowed by the degree of undercooling to experience metastable phase separation. Those droplets receiving sufficient undercooling to additionally penetrate the glass transition can be thermodynamically analyzed to explore the nature of nucleation in phase-separated amorphous materials. Primary analysis tools of the processing include secondary electron emission microscopy (scanning electron microscope), X-ray diffraction, TEM and electron microdiffraction, and differential thermal analysis and scanning calorimetry. These data should allow discrimination of the role of amorphous-amorphous interfaces on crystalline nucleation in the phase-separated amorphous droplets. The study will be extended to the binary  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  system, the alumina-rich end producing highly inviscid melts that, if sufficiently undercooled, could produce interesting alumina/mullite glass-ceramics.

#### TASK SIGNIFICANCE:

Both of the specific tasks in this research are critically dependent on containerless processing: in both cases, avoiding containers eliminates the most blatant source of heterogeneity's that could promote heterogeneous nucleation. In the case of redox reactions (Task 1) the containerless requirement is additionally (and particularly) important in that chemical (as opposed to structural, e.g., nucleation reactions can grossly affect the ionic-scale dynamics and structure of a transition metal-bearing melt. For example, noble-metal crucibles (e.g. platinum) that are often employed to contain refractory ionic melts will alloy with the transition metal ions incorporated in the melt: chemical diffusion different from that desired in the redox experimental results.

Microgravity comes into play when working with inviscid ceramic melts, specifically in the same two manners cited for dealing with molten metals : (1) density contrast amongst phases and (2) the need for quiescence. Both of these aspects are evident in our levitated melt drop experiments: melt viscosities of approximately 1 Pa.s (10 Poises) allow for shear-force-induced convection (droplets are far too small for thermal convection at this viscosity; processing of modestly larger melt bodies could promote thermal convection), which allows continuous exposure of new melt to the outside atmosphere thus short-circuiting the desired chemical diffusion process; in droplets avoiding convection, Ostwald ripening of internally formed ferrites allows them to sink to the bottom of the droplet, thus removing them from positions to act as internal nuclei for silicate phases.

#### PROGRESS DURING FY 1994:

##### 1. Nucleation by internal oxidation or reduction of transition metal-bearing silicate glasses and melts.

Oxidation experiments were completed on a suite of  $\text{CaO-Na}_2\text{O-FeO-MgO-Al}_2\text{O}_3\text{-SiO}_2$  glasses (prepared from naturally occurring Columbia River Plateau Basalts) and on synthetic compositions minus  $\text{Na}_2\text{O}$ . These experiments were analyzed by Rutherford backscattering spectroscopy (RBS), optical microscopy, transmitted electron microscopy (TEM) and high energy electron diffraction (HEED). The RBS spectra clearly indicate that the oxidation process is accompanied by the flux of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  (both specimen types) and  $\text{Na}^+$  (natural basalt specimens) to the free surface with the subsequent formation of a two phase mixture of crystalline  $\text{CaO}$  and  $\text{MgO}$  that *partially covers the specimen surface*. Beneath this surface layer lies a depleted silicate glass, that includes fine precipitates of  $\text{MgFe}_2\text{O}_4$  spinel. The morphology of the reaction is consistent with the oxidation occurring by cation diffusion out of the glass. The kinetics are parabolic (diffusion limited); the rate-limiting step for the oxidation/spinel nucleation process is thus diffusion of the divalent cations away from the reaction front towards the free surface. The experiments have been successfully extended to melts in two ways. Spheres of both the natural and synthetic compositions have been aero-acoustically levitated (AAL) and oxidized above the liquidus for the original melt composition. In the spheres so levitated that *remained quiescent*, the internal oxidation/nucleation process is plainly evident: internal homogeneous nucleation of magnesioferrite spinel (as well as Ostwald ripening of these nuclei) and heterogeneous nucleation (on the spinel precipitates) of calcium -rich feldspar occurred. In similar experiments employing electrostatic levitation (ESL), the synthetic specimens were successfully levitated. Because of the limited capabilities of controlling the atmosphere in the ESL apparatus, a reduction reaction was promoted thermally. The process results in nucleation of pyroxenes on the specimen surface (on Fe metal nuclei) and an inward flux of metallic iron to so promote internal nucleation of silicates. Immediate future plans are to finish the development of the kinetic models to describe these phenomena and to characterize, via TEM and HEED, the crystalline nucleation process that occurs at the reaction front. Longer term plans are the transfer of the idea from silicates to non-glass-forming oxides.

**2. Internal nucleation of inviscid pseudobinary silicate melts via metastable liquid-phase immiscibility.**

MgO-SiO<sub>2</sub> binary melts (metasilicate composition) have been drop-tube processed at approximately 1800°C. Yield of these experiments was surprisingly extensive: approximately 80 vol.% of the fine (<50-μm particles experienced melting in the process (a substantial increase in yield over powder processed at ~1600°C). We are presently collecting statistics on this processed powder, specifically discerning the amount of material that received sufficient undercooling so as to remain amorphous. We are further pursuing thermal analysis experiments on the amorphous powder to ascertain, if possible, the role of amorphous phase separation on the nucleation of glass-ceramic (i.e., spatially uniform, fine-grained crystalline) microstructure in these droplets. Time resolved thermal analysis should allow discrimination of surface nucleation processes (i.e., heterogeneous nucleation on amorphous-amorphous interfaces) from possible homogeneous nucleation processes, as well as determine the scale of phase separation important to promote appropriate crystallization. We anticipate collecting such dynamic data in the next few months. Future plans include the extension of the work to a more inviscid system, Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>, which additionally has significant economic value (i.e. mullite ceramics) with successful melt -processing to a glass-ceramic microstructure.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	2
PhD Students:	1	PhD Degrees:	0

TASK INITIATION: 12/91    EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-26-07-01

RESPONSIBLE CENTER: JPL

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Cooper, R.F. "Glass formation and nucleation in microgravity containerless-processed, inviscid silicate/oxide melts: ground-based studies." MSAD Conference, Huntsville, AL, May 1994.

Directional Solidification in  $^3\text{He}$ - $^4\text{He}$  Alloys

PRINCIPAL INVESTIGATOR: Prof. Arnold Dahm

Case Western Reserve University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The goal of this research is to enhance our fundamental understanding of crystal growth kinetics, liquid-solid interface morphologies, and the stability of alloys.

**TASK DESCRIPTION:**

A study of dendritic growth in two-dimensional samples of  $^4\text{He}$  is underway. Systematic studies of directional solidification in  $^3\text{He}$ - $^4\text{He}$  alloys will be conducted to complement work that has been done in other alloys. Heat conduction plays a minor role in this alloy at low  $^3\text{He}$  concentrations, and the role of heat and particle transport can be isolated.

**TASK SIGNIFICANCE:**

The study of morphologies is of intrinsic interest in testing theories of non-linear systems. Results which differ from those of classical alloys will guide both theorists and experiments in their future studies, and significant differences should result in new ideas for space-based alloy growth experiments. The studied  $^3\text{He}$  -  $^4\text{He}$  transparent alloys have the advantage that they can be studied in a regime where the effects of concentration gradients can be isolated from thermal gradients. That allows for a more complete understanding of the process of alloy solidification and enhance the ability to design tailored materials (alloys with specific material properties).

**PROGRESS DURING FY 1994:**

The goal of this research is to observe directional solidification in  $^3\text{He}$  -  $^4\text{He}$  mixtures in the temperature range near 1.4 K where the melting curves for different concentrations of  $^3\text{He}$  cross. Directional solidification should occur when the solute rich phase freezes at a higher pressure, i.e., fluctuations at the interface leading to regions which are rich in  $^3\text{He}$  will remain fluid, and the concentration in these regions will increase as the  $^3\text{He}$  isotope is expelled from the advancing solid fingers around these regions. When the solute freezes at a lower pressure, regions which are rich in  $^3\text{He}$  will freeze first, and  $^3\text{He}$  will be expelled into regions of depleted  $^3\text{He}$  leading to a restoration of a flat interface. Thus, regimes where fingering does and does not occur should be separated by a small temperature difference.

We have built an experimental apparatus for growing solid helium in a cell with visual access. A new technique for sealing cells with large (2.5 x 5 cm) glass windows at 30 atm has been developed. The apparatus has undergone numerous alterations as unforeseen problems have been solved.

In our experiment solidification is driven by a pressure differential at constant temperature. Solidification occurs at pressures  $\geq 25$  atm. Our initial attempts to observe directional solidification led to some unforeseen difficulties. Solid helium is not supposed to wet metals. However, on increasing the pressure, the solid phase grew from the metallic sides of the cell instead of advancing from the horizontal solid-liquid interface in the cell. In addition, the fill capillary would block when solid wet the walls of the capillary. We believe that these problems were associated with the excellent heat conductivity of the metal. We have solved the problem of capillary blockage by insulating the capillary from the cold helium. We have built a epoxy cell with a copper strip in the bottom so that the heat cannot flow out of the sides of the cell. Recently, one of our cell exploded and broke our dewar. We are installing a new dewar, and experimentation on directional solidification will resume soon.

We have observed dendritic growth in two dimensions in pure  $^4\text{He}$ . To our knowledge this is the first two-dimensional dendrite experiment. The dendritic tips are of dimensions of 1 - 3 mm, while our cell thickness is 0.5 mm. Thus, heat flow is restricted to a plane, and the boundary conditions on equations describing two-dimensional dendritic growth differ from the three-dimensional case. Control of the supersaturation temperature is difficult in this case.

Our program is to first study dendritic growth in two dimensions. This will not only be a new system for investigation, but will give us experience for the directional solidification experiment. Next, we will undertake a systematic study of directional solidification as a function of temperature and  $^3\text{He}$  concentration.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

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TASK INITIATION: 1/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-25-05-25

NASA CONTRACT No.: NAG3-1412

RESPONSIBLE CENTER: LeRC

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*Advanced Photonic Materials Produced by Containerless Processing*

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PRINCIPAL INVESTIGATOR: Dr. Delbert E. Day

University of Missouri, Rolla

CO-INVESTIGATORS:

C.S. Ray

University of Missouri, Rolla

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**TASK OBJECTIVE:**

The objectives of this research were to 1) investigate non-linear optical glasses which have the potential for use as ultra-fast, all optical switches and other photonic devices for communication and advanced computer application, and 2) investigate and compare the kinetics of nucleation and crystallization for these glasses prepared by containerless melting with the crystallization kinetics for the same glasses conventionally melted in a container.

Glasses with the potential for non-linear optical (NLO) applications such as the heavy metal oxide (HMO) glasses containing PbO, Bi<sub>2</sub>O<sub>3</sub> and Ga<sub>2</sub>O<sub>3</sub> are, in general, highly fluid and chemically corrosive. These melts readily crystallize during cooling and develop unwanted color centers primarily due to impurities dissolved from the container. The traditional procedures used to melt these glasses yield colored and chemically inhomogeneous glasses of limited usefulness for NLO applications.

Containerless melting provides the opportunity to suppress or eliminate the undesirable heterogeneous nucleation and crystallization in such melts. Since no container is used, color centers caused by impurities dissolved from a container can be completely eliminated, even in highly corrosive melts. Containerless processing offers a viable alternative for preparing glasses with improved purity and homogeneity, and NLO properties.

**TASK DESCRIPTION:**

Investigations of the NLO glasses are to be conducted with two HMO compositional systems: (1) PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> and (2) compositions based on TeO<sub>2</sub>. The importance of the PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses for NLO applications have been demonstrated by scientists at Corning Inc., but these glasses had a color ranging from orange to yellow which is believed to have come from impurities dissolved from the container. Attempts will be made to obtain colorless PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses by changing the melting parameters that generally affect the color of a glass such as the crucible material, melting temperature, time and atmosphere, and the starting raw materials. Containerless melting technique will also be used to process these glasses so as to eliminate the color centers that are developed from the impurities dissolved into melt from a container.

Like the PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses, the tellurite glasses also have a high refractive index ( $>1.9$ ) along with a low Abbe number (10 to 20), or high dispersion. Calculations using the empirical equation,  $n_2 = 391(n_d - 1)/V_d^{5/4}$  which was developed by Boling et al. (N. L. Boling, A. J. Glass and A. Owyong, IEEE J. Quantum Electronics, QE-14, 601, 1978) and widely used by others, indicate that the tellurite glasses have a non-linear refractive index,  $n_2$ , even larger than that of the PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses ( $n_d$  and  $V_d$  are the usual linear refractive index and Abbe number, respectively, for the glass with respect to sodium D-light). Also tellurite glasses have excellent optical transmission ( $>85\%$ ) over a wide range, from 400 to 3500 nm. These make the tellurite glasses promising laser hosts for a variety of rare earth ions, including Nd<sup>3+</sup> (primary laser wavelength 1060 nm).

The tellurite glasses for NLO applications will contain oxides of heavy metals such as Nb, Ta, Pb, and Bi to ensure high density and refractive index (linear). Generally, glasses with a high refractive index also have a non-linear refractive index (see the equation above). Minor addition of the oxides of Zn, Ga, Ca, Al and Ba in these glasses will also be considered if needed to improve glass formation. The following work will be performed for both type of HMO glasses.

- (1) Measure critical cooling rate of glass formation,  $R_c$  (container), using the pendant drop technique (spherical glass melt hanging from a thermocouple bead) to determine the glass forming tendency for these melts.
- (2) Use containerless melting techniques to prepare the glasses if  $R_c$  (container) for these glasses appear to be very high and/or contamination of the melts with the container melts is significant.
- (3) Measure and compare  $R_c$  (containerless) and  $R_c$  (container). The ratio of  $R_c$  (container) to  $R_c$  (containerless) will be used to determine the improvement in glass formation for containerless melts at 1-g. The  $R_c$  (containerless) will be measured at CRI, Inc., using their aero-acoustic levitator furnace.
- (4) Measure selected properties such as the density, molar volume, linear refractive index and Abbe number, chemical durability, thermal expansion coefficient, glass transition and crystallization temperatures, and the transmission in the visible-ultraviolet and IR to determine the suitability of these glasses in practical applications.
- (5) Calculate the non-linear refractive index,  $n_2$ , for each glass from the equation  $n_2 = 391(n_d - 1)/V_d^{5/4}$  and identify those compositions best suited for NLO applications.
- (6) Investigate and compare the kinetics of nucleation and crystallization for these glasses prepared by traditional method and by containerless fashion.
- (7) Measure XRD, IR and Raman spectra and evaluate the structure of these glasses.

**TASK SIGNIFICANCE:**

The following results are anticipated from this ground based investigation.

- (1) The effect of different cations on the NLO properties of these glasses will be understood. This result will be useful to developing glasses that have the NLO properties superior to those presently available.
- (2) Comparing the properties for the glasses prepared by the traditional method with those for the glass prepared by containerless melting, the need for processing these glasses in the microgravity environment of space for achieving improved NLO properties will be known. However, the absence of gravity driven convection on the NLO properties for these glasses will remain unknown. The results from several space-borne (low gravity) glass melting experiments indicate that a glass prepared in microgravity is more resistant to crystallization and more chemically homogeneous. The optical properties including the NLO and lasing efficiency for a glass processed in microgravity are, therefore, expected to be improved compared to those for a glass at 1-g.
- (3) The physical, thermal, optical, and spectroscopic properties for these HMO glasses that have the potential for NLO and laser applications will be known for the first time. The kinetics of nucleation and crystallization for these glasses along with their tendency towards glass formation will also be known.

**PROGRESS DURING FY 1994:****A. PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> Glasses**

1. IR Spectra: To evaluate the structure of these PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses, the IR spectra from 400 to 3500 cm<sup>-1</sup> were measured for 14 glasses as a function of composition using the KBr pellet technique. The IR spectra of these glasses, generally have the same broad peak between 542 to 555 cm<sup>-1</sup> which is identified as the vibration of Ga-O in GaO<sub>4</sub> groups. This result suggests that Ga<sup>3+</sup> ions act primarily as network forming cations in these glasses. When Ga<sub>2</sub>O<sub>3</sub> exceeds about 30 mol%, a new peak at about 730 cm<sup>-1</sup> appears in the IR spectra, which is due to the vibration of Ga-O in GaO<sub>6</sub> groups. Thus, a part of the Ga<sup>3+</sup> ions appear to be in 6-fold coordination when the Ga<sub>2</sub>O<sub>3</sub> content exceeds 30 mol% in these glasses.

2. Raman Spectra: To further investigate the structure of these PbO-Bi<sub>2</sub>O<sub>3</sub>-Ga<sub>2</sub>O<sub>3</sub> glasses, it was planned to measure the Raman spectra of the glasses and their devitrified products as a function of composition. These measurements were undertaken by Dr. A. Biswas at JPL using samples provided by UMR. While these

measurements are presently continuing, measurements of Raman spectra for a  $20\text{PbO}.60\text{BiO}_{1.5}.20\text{GaO}_{1.5}$ , cat%, glass (HMO-1) and its crystallized products have been completed. The crystallization of the HMO-1 glass was performed at  $420^\circ$ ,  $480^\circ$ ,  $500^\circ$ , and  $575^\circ\text{C}$  for 1 h at each temperature. These data are now being analyzed, but the preliminary results suggest the presence of Bi-O vibrational bonds, possibly in the  $\text{BiO}_6$  groups, in all the samples crystallized at  $420^\circ$ ,  $480^\circ$ , and  $500^\circ\text{C}$ . The peaks corresponding to the vibration of Bi-O bond appeared in the Raman spectra, most likely, for the crystallization of  $2\text{PbO}.3\text{Bi}_2\text{O}_3$  crystals. These crystals were identified by XRD when this HMO-1 glass was crystallized at  $420^\circ$ ,  $480^\circ$ , and  $500^\circ\text{C}$ . No peak corresponding to the vibration of Pb-O appeared in the Raman spectra of these samples. The PbO bond in the  $2\text{PbO}.3\text{Bi}_2\text{O}_3$  crystals is mostly ionic and, hence, too weak to be detected by Raman measurements. New peaks corresponding to the vibration of Pb-O appeared in the Raman spectra for the sample crystallized at  $575^\circ\text{C}$ . These Pb-O bonds are stronger and perhaps more covalent like the bonds in the  $\text{PbO}_4$  groups present in  $\text{PbO}_2$  crystals. This result from the Raman spectra confirms the XRD results obtained previously for the HMO-1 glass heat treated at  $575^\circ\text{C}$  for 1 h, where  $2\text{PbO}_2.12\text{Bi}_2\text{O}_3$  crystals were identified.

3. Nucleation Rate Curve: Except for the glasses containing a high amount of PbO (50-60, cat%), all the glasses investigated in this  $\text{PbO}-\text{Bi}_2\text{O}_3-\text{Ga}_2\text{O}_3$  system yield multiple DTA crystallization peaks, which makes it difficult to determine either the activation energy for the crystallization peaks or the relative rates of nucleation at different temperatures for these glasses. The activation energy for crystallization for a  $60\text{PbO}.10\text{BiO}_{1.5}.30\text{GaO}_{1.5}$ , cat% (HMO-12) glass, which contains a single DTA crystallization peak, was determined and was  $(470 \pm 15 \text{ kJ/mol})$ . The same HMO-12 glass was used to determine a nucleation rate type curve using the DTA technique developed by ourselves (Ray and Day, J. Amer. Ceram. Soc. 73, 439, 1990). In this technique, glass samples of constant weight and particle size are first heated isothermally in the DTA furnace for a fixed time (nucleation), and then heated at a constant rate until the glass crystallizes (crystallization). A plot of either the maximum height of DTA crystallization peak, (dTp) or the inverse temperature at the peak height maximum,  $1/T_p$ , as a function of nucleation temperature yields a curve similar to the well known nucleation rate vs. temperature curve found in many silicate glasses. It was also shown that the dependence of dTp on nucleation temperature was more sensitive than that of  $1/T_p$ .

When measured and plotted as a function of nucleation temperature, the dTp for this glass does not change from that of the as quenched glass until the glass is nucleated above  $\sim 265^\circ\text{C}$ , it then reaches a maxima at  $\sim 343^\circ\text{C}$  and then decreases to the value for the as-quenched glass at  $375^\circ\text{C}$ . In other words, the temperature for nucleating this glass ranges from  $\sim 265^\circ$  to  $375^\circ\text{C}$  and the temperature where the nucleating rate is a maximum, is  $343^\circ\text{C}$ .

No information on the temperature range for nucleation or the temperature for maximum nucleation rate for this glass is obtained from the plot of DTA peak temperature. In fact,  $T_p$  for a majority of glass samples nucleated at different temperatures lies within the  $\pm 1.5^\circ\text{C}$  limit of experimental error. Instead of yielding a bell-shaped curve,  $T_p$  shows an increasing trend, although slightly, with increasing nucleating temperature, from  $\sim 456^\circ\text{C}$  for the as-quenched glass to  $\sim 464^\circ\text{C}$  for the glass nucleated at  $395^\circ\text{C}$ . The  $T_p$  for this HMO-12 glass measured after different nucleation treatment is, therefore, considered random and independent of the nucleation temperature

## B. Tellurite Glasses:

Investigations on the formation, crystallization, and structure of binary  $\text{Na}_2\text{O}-\text{TeO}_2$  glasses as a function of composition have been started using differential scanning calorimetry (DSC), X-ray diffraction (XRD) and IR spectra. The objectives of this work are to gain a better understanding of (1) glass formation and crystallization kinetics of tellurite melts and (2) the structural role of the Te<sup>-</sup> ions in these glasses. This knowledge will be useful in developing and controlling the composition of  $\text{TeO}_2$  containing glasses that have potential for non-linear optical (NLO) and laser applications.

For DSC measurements, about 20 mg of glass powder composed of  $\sim 100 \mu\text{m}$  particles was sealed in an aluminum pan, which was then heated at  $10^\circ\text{C/min}$ . in an atmosphere of flowing nitrogen until crystallization was complete. The DSC crystallization patterns for five glasses, with compositions  $\text{Na}_2\text{O}.2\text{TeO}_2$  ( $\text{NT}_2$ ),  $\text{Na}_2\text{O}.3\text{TeO}_2$  ( $\text{NT}_3$ ),



$\text{Na}_2\text{O} \cdot 0.4\text{TeO}_2$  ( $\text{NT}_4$ ),  $\text{Na}_2\text{O} \cdot 0.5\text{TeO}_2$  ( $\text{NT}_5$ ), and  $\text{Na}_2\text{O} \cdot 0.6\text{TeO}_2$  ( $\text{NT}_6$ ) have been measured to date. With the exception of the  $\text{NT}_4$  glass, all the other glasses exhibit multiple crystallization peaks, indicating crystallization of two or more phases. The onset temperature for crystallization is highest for  $\text{NT}_4$  glass indicating this glass is most stable of those measured. The activation energy for crystallization and a nucleation rate type curve for this  $\text{NT}_4$  glass are now being measured using DSC. Sodium tellurite glasses were crystallized at their respective maximum temperatures (obtained from DSC) for 24 h and analyzed by XRD. The crystalline phase(s) formed in each glass are in accordance with the phase diagram for this binary  $\text{Na}_2\text{O}$ - $\text{TeO}_2$  system, with the exception that in the devitrified  $\text{NT}_3$  glass both  $\text{NT}_4$  and  $\text{NT}_2$  crystals should be present according to the phase diagram, while only  $\text{NT}_4$  crystals were identified in this sample. A comparison of the number of crystalline phases identified by XRD (only 1 or 2 for all the compositions) with the multiple crystallization peaks exhibited by DSC suggests that these glasses undergo a series of phase transformations during heating.

The IR spectra for these sodium tellurite glasses and their devitrified counterparts have been measured using the KBr pellet technique. Preliminary analysis show that the structure of the  $\text{NT}_4$  glass contains primarily  $[\text{TeO}_4]$  bipyramidal groups, while the structure of all the other glasses of higher or lower  $\text{TeO}_2$  content than the  $\text{NT}_4$  glass, contain both  $[\text{TeO}_3]$  pyramidal and  $[\text{TeO}_4]$  bipyramidal groups. Further analysis of the IR spectra for these glasses is continuing.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/91 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-26-07-02

RESPONSIBLE CENTER: JPL

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*The Effect of Gravity on Natural Convection and Crystal Growth*

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PRINCIPAL INVESTIGATOR: Dr. Graham D. de Vahl Davis

University of New South Wales

CO-INVESTIGATORS:

H.C. deGroh III

NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE:**

1. To experimentally measure natural convection during Bridgman growth at varying gravity-driven levels and to quantitatively determine how this convection affects parameters of critical importance to the crystal grower such as interface shape, and radial and longitudinal segregation.
2. To produce an accessible, experimentally verified numerical code capable of accurately determining levels of convection in real systems at varying gravity levels and directions, and the effects of this convection on the solidification process.
3. To supply and supplement numerical modeling efforts for the MEPHISTO II flight experiment of Dr. R. Abbaschian.

**TASK DESCRIPTION:**

The major goal of many space experiments in solidification is to determine how convection influences various phenomena such as undercooling at the solid/liquid interface, redistribution of solute, and crystal quality. However, in most flight experiments, as in the MEPHISTO experiments which are being done using an opaque alloy of Bi-Sn, no measurements of convection are possible. Experiments and numerical studies are being done to estimate convection and its effects. Two codes are under development: 1. A modified version of the commercial finite element code FIDAP (due to M. Yao), and 2. A finite difference code (due to G. de Vahl Davis, E. Leonardi and students). Both may be available to investigators. In our experiments, we are using the transparent metal analog succinonitrile, which allows direct analysis of convection and interface shape.

**TASK SIGNIFICANCE:**

Our research will yield experimentally verified codes capable of calculating amounts of convection present in the melt at 1-g horizontal, vertical, and various intermediate angles, and in low-g with various residual gravity values, angles and frequencies. Thus it is hoped that current flight experiments, as well as future flight experiments, will benefit from this research. The models will complement the scaling, analytical modeling and parametric experimental studies of others (Coriell, Favier, Thevenard and Camel) and will be used to check the validity, extension and generalization of scaling laws.

**PROGRESS DURING FY 1994:**

Solidification Experiments: Experimental studies of succinonitrile during solidification, melting and no-growth conditions using a horizontal Bridgman furnace and square glass ampoules have been conducted. For use as input boundary conditions to the numerical codes, thermal profiles inside and on the outside of the ampoule were measured. The shapes of the s/l interface in various 2-D planes were quantitatively determined. Though interfaces were non-dendritic and non-cellular, they were not "flat," but were highly curved and symmetric in the vertical, longitudinal plane. The shape of the interfaces were dominated by the primary longitudinal flow cell characteristic of shallow cavity flow in horizontal Bridgman; this flow cell was driven by the imposed furnace temperature gradient and caused a "radial" thermal gradient such that the upper half of the ampoule was hotter than the bottom half. These data will be used to examine the numerical models. Presently underway is an effort to measure fluid flow velocities and interface shape and convective modes at various ampoule angles between vertical and horizontal.

Numerical Modeling: The results of the numerical codes have been compared to the experiments in succinonitrile and excellent agreement has been achieved. The codes have been used to predict interface shape, thermal fields and fluid flow velocities during horizontal Bridgman no-growth, and solidification conditions in 2-D and 3-D. The segregated solution approach has enabled fine mesh 3-D solidification simulations, using FIDAP, to be run on a work station. The front tracking technique and solute concentration have been incorporated into the finite element model. In contrast to the formerly used enthalpy-type method, the front tracking technique involves a deforming spatial grid and is able to model phase changes with sharp interfaces. These new efforts have greatly enhanced the capability of our numerical simulations. Using the front tracking method, a 2-D simulation of the capability of our numerical simulations. Using the front tracking method, a 2-D simulation of the MEPHISTO II vertical 1-g and space experiments has been done; it was found at  $g < 10^{-4}$  earth gravity, solidification was diffusion controlled. 3-D finite difference solution methods using body-fitted coordinates have allowed an accurate representation of the interface shape, position and growth to be obtained, together with detailed results for the flow pattern and temperature distribution. Other low-g simulations are underway and future efforts will include non-steady gravities (g-jitter) and with g at various angles to the ampoule axis, as is expected in space.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 11/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 962-21-05-09

RESPONSIBLE CENTER: LcRC

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### *Use of Synchrotron White Beam X-ray Topography for the Characterization of the Microstructural Development of Crystal - Normal Gravity Versus Microgravity*

PRINCIPAL INVESTIGATOR: Dr. Michael Dudley

State University of New York, Stony Brook

CO-INVESTIGATORS:

D. Larson

Northrop-Grumman

#### TASK OBJECTIVE:

The objective of the research proposed here is to provide an assessment of the influence of the accelerated cooling rates, imposed by severe limitations on available flight time, on the defect microstructure of crystals grown in a microgravity environment (using, for example, modified Bridgman or Vapor Transport techniques). Results previously obtained on ground based ZnTe samples (grown by Vapor Transport at NASA Marshall Space Flight Center) seem to indicate that if cooling rates are too high the accentuated thermal mismatch stresses can give rise to deformation processes, comprising the formation of dislocation slip bands.

The objective of the present research is to extend the preliminary work on ground based samples, to further ground based samples and also to flight samples grown with different cooling rates. The microstructures of the as-grown crystals will be non-destructively characterized using the technique of Synchrotron White Beam X-ray Topography (SWBXT). Correlations between the existence of significant post-growth deformation and accelerated growth rates will be directly examined.

The influence of accelerated cooling rates on the significance of the comparison drawn between normal gravity and microgravity crystal growth can thus be determined.

#### TASK DESCRIPTION:

The research proposed here will be carried out using Synchrotron White Beam X-ray Topography (SWBXT) at the Stony Brook Synchrotron Topography Facility, with is under the direction of Prof. Dudley. It will consist of SWBXT characterization of crystals grown in a microgravity environment primarily obtained from co-investigator Dr. D.J. Larson, Jr., of Grumman Corporate Research Center, as well as from other sources such as Drs. D. Gillies, and C. Su at NASA Marshall Space Flight Center. Crystals of CdZnTe and ZnTe will be examined, although many other systems of interest to NASA are expected to be studied, for example ZnSe, CdTe, HgCdTe and PbSnSe. Opportunities will be sought amongst the NASA crystal growth community to maximize the amount of data obtained on as many different systems as possible. Crystals grown at various cooling rates will be examined, and the influence of these rates on the resulting microstructure will be assessed.

In the approach adopted here, reflection topographs from the cylindrical outside surface of the as-grown boules will be initially examined. These will provide information on the defect structures in the region from the outside surface down to the penetration depth of the X-ray beam, which is typically 5-10 m. This enables an overall assessment of the distribution of defects such as twins, sub-grain and grain boundaries, and dislocations to be made. This can aid in the development of optimal wafering geometries to enable clearest visualization of the defect microstructure. Following this, both reflection and transmission topographs will be recorded from the individual wafers. Images obtained from the various wafers will be compared with those obtained from the original boules, and an overall representation of the three dimensional distribution of defects throughout the boule will thus be developed. By comparing such three dimensional representations of defects in crystals grown with various cooling rates, the influence of the cooling rate on the type and distribution of defects in the crystals can be determined.

It is anticipated that results may indicate that accelerated cooling rates, dictated by schedule limitations, have a dominant effect on the microstructure of crystals grown in a microgravity environment, thus obscuring information

on the influence of the magnitude of the gravity vector. It is further anticipated that these results may lead to a change in the criteria used to determine time allocation for individual crystal growth experiments on future flights in order to avoid effects on crystal microstructure resulting from effects unrelated to the magnitude of the gravity vector.

#### TASK SIGNIFICANCE:

Much effort has been, and continues to be expended by NASA in evaluating the influence of a microgravity environment on the defect microstructures developed in crystals during crystal growth. While the influence of a microgravity environment on crystal growth is generally accepted as being beneficial, it is not clear that prior studies of this influence have been unperturbed by artifacts related to the particular choice of experimental conditions. Indeed, in order to properly compare crystal growth in a microgravity environment to that in a normal gravity environment it is important to be able to isolate the influence exerted by the difference in magnitude of the gravity vector on the resultant crystal quality. Effects associated with accelerated cooling rates, or sudden changes in temperature gradient, imposed either deliberately or inadvertently, detract from the significance of such experiments. Results already obtained from ZnTe crystals grown in a normal gravity environment seem to indicate that stresses generated by thermal mismatch effects during cooling can significantly affect the observed, as-grown defect microstructure. These stresses lead to the formation of dislocation slip bands which tend to obscure and/or break up the true growth defect microstructure. Should such effects occur in crystals grown in a microgravity environment, the true influence of microgravity on crystal quality cannot be assessed.

It is thus of paramount importance to be able to assess the effect of the accelerated cooling rates, dictated by flight schedule limitations, on resulting defect microstructures. One can gain insight into the chronological history of the development of the final defect microstructure from analysis of images recorded using Synchrotron White beam X-ray Topography (SWBXT). This is a non-destructive imaging diffraction technique capable of revealing the detailed defect microstructure in large single crystals. Such defect microstructures in crystals grown with various cooling rates will be compared and examined for evidence of extensive, post-growth slip. Maximum usable cooling rates can thus be determined.

Determination of the influence of cooling rate on the defect microstructure of crystals is crucial for selection of experimental conditions under which the effects of the gravity vector on crystal growth quality can be usefully investigated. Once such selection has been optimized, differences in microstructure observed in microgravity grown crystals may be safely attributed to the influence of the gravity vector, and not to artifacts related to compressed growth schedules.

Should accelerated cooling rates be shown to have a detrimental effect on defect microstructures in microgravity grown crystals, it is likely that this result could modify the criteria used to determine the experimental schedules for microgravity crystal growth.

#### PROGRESS DURING FY 1994:

Significant progress has already been made on this project. Professor Dudley, Dr. Larson, and Graduate student Hua Chung have been systematically recording topographs from several wafers cut from three boules: one ground based CdZnTe sample and two flight samples (numbered 1 and 2), all grown by Dr. D.J. Larson, Jr. (the latter two on USML-1). These crystals were originally examined in boule form using reflection topography. This revealed the overall distribution of defects in the outer cylindrical surface of the boule down to a depth of around 5-10 micrometers. Twins and slip bands were readily revealed. Twins appeared to nucleate at regions of partial wall contact. Further studies of the effects of cooling rate on microstructure are envisaged in as-grown boules, prior to cutting. As in the case presented below this will be followed up with studies of as-cut wafers. The ability to investigate boules, as-grown, before cutting, means that one can be sure that one is observing the true microstructure developed during the growth process, with no possibility for artifacts, for example due to polishing or cutting, to exist. In addition, if effects due to thermal mismatch associated with rapid cooling are of interest, these are likely to give rise to deformation microstructures which would be concentrated near the periphery of the boule, which is precisely the volume examined in these experiments.

Topographic images recorded from wafers sliced from the ground-based sample showed slip bands and a relatively high dislocation density (in excess of  $105\text{cm}^{-2}$ ). Examination of wafers cut from the most slowly cooled region of flight sample number 2 reveals very low dislocation density regions (less than  $102\text{cm}^{-2}$ ) with individual dislocations being easily resolvable. A low density of precipitates is also clearly observed. Images recorded from a more rapidly cooled region of the same sample shows higher dislocation density (approaching  $104\text{cm}^{-2}$ ). Topographs recorded from a wafer sliced from flight sample number 1 which was more rapidly cooled than flight sample number 2 reveal a significantly higher overall dislocation density (approaching  $105\text{cm}^{-2}$ ) and also the existence of extensive slip which is observable throughout the imaged volume. These results confirm the initial hypothesis that accelerated cooling rates, imposed by compressed flight schedules, could potentially obscure and/or destroy the underlying actual growth defect structure, hindering the assessment of the influence of gravity vector magnitude on crystal quality. In general it appears that increasing cooling rate first increases the overall growth dislocation density, and then produces slip which obscures this growth defect structure. Further detailed work is underway to examine more wafers from these boules, and from other boules, in order to further examine this correlation between the observed three-dimensional defect structure and the local cooling rate.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-21-08-27

NASA CONTRACT No.: NCC8-48

RESPONSIBLE CENTER: MSFC

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Dudley, M. "White beam synchrotron topography of II-VI crystals." Invited lecture at Loral Infrared and Imaging Systems, Lexington, MA, December 2, 1993.

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*Reverse Micelle Based Synthesis of Microporous Materials in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Prabir K. Dutta

Ohio State University

## CO-INVESTIGATORS:

Dr. C.T. Kresge

Dr. R. Ansari

W. Meyer

Mobil Research &amp; Development Corporation

NASA Lewis Research Center (LeRC)

NASA Lewis Research Center (LeRC)

## TASK OBJECTIVE:

The objective of this research is to better understand the synthesis of microporous materials, especially those based on a network of connecting substructures. Examples of microporous materials include silico-aluminates widely used as catalysts and absorbents and natural bio-materials such as bone.

## TASK DESCRIPTION:

The synthesis of zincophosphate microporous material is studied via the novel synthesis route of reverse micelles. The reverse micelles isolate small portions of reactant and allow examination of different fabrication routes emphasizing single layer additive growth, agglomeration of subsize particles and reconstruction from a gel-like mass.

The different paths are studied to determine the influence of convective flows and Stokes settling. Conditions for zincophosphate growth appropriate for on orbit experimentation are identified.

## TASK SIGNIFICANCE:

Microporous materials have extremely wide application as catalysts and absorbents. Their complex structure is derived from chemical and physical transformations which have to date eluded investigators. This research separates portions of the nucleation and growth process for examinations. It may lead to greater understanding of how these materials form, to new synthesis routes, to new materials and thereby to changes economics of petroleum processing.

## PROGRESS DURING FY 1994:

Three paths for zincophosphate crystal growth have been identified. These paths involve benign using reverse micelles procedures appropriate for performance on orbit. It has been shown by tests using rotating cells that the synthesis is sensitive to Stokes settling. It has also been shown that laser light scattering tracks particle growth at sizes larger than the predominant micelles size.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 963-26-05-01

NASA CONTRACT NO.: NAG3-1416

RESPONSIBLE CENTER: LeRC

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**Presentations**

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*Growth of Nonlinear Optical Thin Films and Single Crystals by Vapor Processes*

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PRINCIPAL INVESTIGATOR: Dr. Donald O. Frazier

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

B. Penn

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

A major goal of this program is to study the dynamics of physical vapor transport for the growth of high-quality thin films and single crystals of nonlinear optical (NLO) organic and polymeric materials. The program involves the experiment and modeling of heat and mass transport during vapor deposition of materials of interest, and subsequent coupling with film formation kinetics, and application to bulk crystal growth.

**TASK DESCRIPTION:**

This work now focuses on specific materials of interest for study. Initially, the program involved the screening and tailoring of many possible derivatives, and included synthesis, purification, processing, and characterization to determine the best possible NLO organic and polymeric materials for microgravity study. The work now focuses on vapor deposition of a 2-methyl -4-nitroaniline (MNA) diacetylene derivative on various substrates, and subsequent solid-state photopolymerization to the corresponding polydiacetylene. Also, the work continues the microgravity experiment to deposit phthalocyanine films (porphyrins, generally), by 3M Corporation, which were dramatically different from Earth-processed thin films. Both of these materials have excellent third-order NLO properties, but detailed information on the gravitational effect on film formation by physical vapor transport is absent. This work studies coupling between heat and mass transport and film/crystal morphologies.

Research Approach - The approach is to prepare thin films of diacetylene monomers and photopolymerize the monomer deposits on various substrates. Deposition of metal-free phthalocyanines also yields films of excellent third-order nonlinearity (also, good second harmonic generation (SHG) is possible). Metal-free phthalocyanines also provide appropriately oriented substrates for deposition of metal phthalocyanines which could enhance electron mobilities and polarizabilities. These experiments are in progress with plans for future microgravity processing to study enhancement of orientation, and modeling of the physical vapor transport process. Fabrication of a horizontal furnace is complete, and physical vapor transport of 4-N,N'-dimethylamino-4'-N-methylsulbazolium tosylate (DAST) will proceed by seeking coupling between heat and mass transport and growth kinetics. DAST, because of the ionic nature of its crystal packings (a salt), could provide the basis for improving the mechanical strength of organics having very high SHG efficiencies.

**TASK SIGNIFICANCE:**

Many of the crystal/film growth processes appropriate for inorganic crystal growth and which amplify key elements of microgravity materials processing are equally applicable to the growth of organic photonic materials. Indeed, there is an opportunity to observe the effects of fundamental microgravity related phenomena not possible to study in semiconductors. Furthermore, these materials may comprise a large share of the next generation of materials for computer applications.

**PROGRESS DURING FY1994:**

This work has advanced from screening and tailoring of many possible derivatives, to vapor deposition of a 2-methyl -4-nitroaniline (MNA) diacetylene derivative on various substrates, and subsequent solid-state photopolymerization to the corresponding polydiacetylene. Additional focus is on the incomplete microgravity experiment to deposit phthalocyanine films (porphyrins, generally), by 3M Corporation, which were dramatically different from Earth-processed thin films. We plan to complete this work by modeling the heat and mass transport,

absent in the initial study, and coupling with the kinetics of film formation. This work is underway and a proposal is in preparation.

We have determined the importance of substrate thermal control during the diacetylene derivative deposition, and accomplished deposition of very good films by physical vapor transport on a gallium arsenide substrate. Along with observation of other systems, substrate thermal control has caused some interesting speculation on the dynamics of film formation during physical vapor transport.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/91 EXPIRATION: 1/94

PROJECT IDENTIFICATION: 962-21-08-12

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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*Growth of Nonlinear Optical Crystals by Melt and Solution Processes*

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PRINCIPAL INVESTIGATOR: Dr. Donald O. Frazier

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

B. Penn

NASA Marshall Space Flight Center (MSFC)

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**TASK OBJECTIVE:**

Our objectives are to study formation of organic nonlinear optical (NLO) bulk composites and amorphous films having excellent nonlinear properties under certain processing conditions. Detailed determinations of probable gravity induced phenomena during processing will provide appropriate microgravity experiment rationale.

Another primary objective is to study dynamics of solution growth using organics, including certain peptides, with good nonlinear optical properties.

**TASK DESCRIPTION:**

The emphasis on organic crystals and thin films has shifted to composites and amorphous materials. This is due to weaknesses in mechanical strength, optical defects, decomposition of pure crystals grown by Bridgman-Stockbarger techniques, and difficulty in achieving large areas of crystallinity in polymerized films. Some of the most promising processing techniques in this area of research now include preparation of bulk composites of the active NLO material in a polymer matrix, physical vapor transport, and the photopolymerization of diacetylene monomers in solution.

A recent objective, therefore, is to assess methods of extending excellent third-order nonlinear optical organics in solution to organics in polymer matrices. For optimal nonlinear properties and good optical quality, composition is critical, and methods for achieving uniform and accurate composition, quite specific. Solution methods include photopolymerization of diacetylene derivatives, which yield amorphous thin films of excellent third-order nonlinearity, as well as future crystal growth of peptides and other organics such as 4-N,N'-dimethylamino-4'-N-methylstilbazolium tosylate (DAST) with very high powder second harmonic generation (SHG) efficiencies.

The photopolymerization technique results in films of a polydiacetylene from a 2-methyl-4-nitroaniline diacetylene derivative from monomer solutions onto UV transparent substrates. This process yields amorphous polydiacetylene films with optical quality superior to that of films grown by standard crystal growth techniques. These films have excellent third-order NLO susceptibilities, and spatial selection of film deposits is simple. Furthermore, there is a compelling opportunity to study fundamental gravity related phenomena due to processing heat transfer dynamics.

Benzil in acetone results in novel third-order nonlinearities such as "beam fanning" and transient multiple diffraction rings. Transiency results from convective dissipation due to electrooptic and/or thermal effects. Dispersion of the active NLO material throughout a polymer matrix, such as polymethylmethacrylate, is stabilizing and of technological interest, however, it is important to optimize bulk concentration and homogeneity of the dispersed phase. The gradient-freeze processing method may introduce gravity-dependent concentration inhomogeneities and effect optical characteristics.

Future aspects of this program include synthesis of peptide derivatives (and DAST) with excellent second-order NLO properties followed by a study directed at understanding processes occurring during the crystal growth of these materials by solution methods. This project would also have value in understanding protein crystal growth dynamics.

**TASK SIGNIFICANCE:**

Many of the crystal/film growth processes appropriate for inorganic crystal growth and which amplify key elements of microgravity materials processing are equally applicable to the growth of organic photonic materials. Indeed, there is an opportunity to observe the effects of fundamental microgravity related phenomena not possible to study in semiconductors. Furthermore, these materials may comprise a large share of the next generation of materials for computer applications.

**PROGRESS DURING FY 1994:**

In our work to study melt growth via the Bridgman-Stockbarger method of directional solidification, we have determined that advantages can be derived from binary systems. Organics with nonlinear optical properties in a solvent exhibits interesting third-order properties. This study was the focus of a Master's Degree and is currently the subject of the incorporation of NLO materials in polymer matrices. These systems exhibit newly discovered properties such as beam fanning and multiple diffraction rings. Dispersion of the NLO active component throughout the polymer following a gradient freeze is the subject of potential microgravity processing.

We have discovered a novel technique for obtaining thin polydiacetylene films using photo-deposition from monomer solutions onto UV transparent substrates. This method, to our knowledge, is unique and offers the opportunity for fundamental microgravity studies and represents a significant advance in the technology of the application of amorphous films having excellent nonlinear optical properties. The microgravity interest can be derived, in part, from the heat transfer dynamics at the container solution interface which result from relative absorption properties of irradiated UV light by the container and the solution. A patent application is in preparation.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 1/91 EXPIRATION: 1/94

PROJECT IDENTIFICATION: 962-21-08-13

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Journals**

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*Investigation of Local Effects on Microstructure Evolution*

PRINCIPAL INVESTIGATOR: Dr. Donald O. Frazier

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

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P. Downey

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B. Facemire

NASA Marshall Space Flight Center (MSFC)

B. Witherow

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The objective of this proposed work is to perform modeling and experimental studies on the development of microstructures and the growth of a second phase in a two-phase system. Most modeling and experimental studies focus on the average particle, and the late stages of growth processes. However, experimental observations of dynamic local behaviors within the context of a particle ensemble are accessible by optical techniques, including holography. Interest extends to examining these systems in all stages of growth within the limit of holographic resolution. Additionally, holography provides and archives data to employ emerging techniques for measuring diffusion fields during growth.

**TASK DESCRIPTION:**

The primary method of data collection is by the use of holography. The holographic techniques are crucial to capturing the entire cell volume. A unique capability to apply phase shifting interferometry to holography is under development by one of the coinvestigators of this work. With this technique it is possible to measure the concentration gradients, hence diffusion fields, surrounding individual droplets. Such data, not attainable by any other techniques, would provide essential input to computer models. Additionally, a two-color holography technique, also under development by the same coinvestigator, may provide measurement of spurious thermal gradients over the lifetime of an experiment. It should be noted that use of narrow path-length cells can introduce the effects of "mixed" dimensionality, as observed in recent analysis of an experiment performed in this laboratory. "Mixed" dimensionality refers to 3-D droplets located on a 2-D substrate. Such systems follow different scaling laws than fully 3-D systems.

The research approach includes observation of diffusional growth of the secondary phase in a transparent monotectic system. Ultimately, this work will rely on the benefits of microgravity processing to eliminate the buoyancy caused by concentration or temperature gradients. Ground work requires a transparent isopycnic system. Sensitivity to gravitational fields is tested, in the laboratory, by tethering two droplets in a test cell and comparing growth kinetics at varying temperatures. Convective flows increase with increasing conjugate phase density differences which are relatively strong functions of temperature. Quench experiments in narrow path-length cells allow observation of local effects in an ensemble of droplets. Narrow path-length is a prerequisite for establishing transparency quickly in high droplet density media.

**TASK SIGNIFICANCE:**

To a large extent, particle growth and distribution determine the mechanical properties of an alloy. An understanding of microstructure is essential to predicting the behavior of a material with respect to, for example, metal fatigue. The unique capabilities provided by holographic techniques enabling observations of local effects during diffusional growth of a second phase in a model, transparent two-phase system, allow detailed analyses of the local dynamic and resulting microstructures. Additionally, holographic studies of droplet diffusion in model systems are in agreement with Rutherford backscattering measurements of Ga and Sn "island" growth on Si. Likewise, cloud dynamics derives some of its physics from such coarsening phenomena. Diffusional studies to model the time evolution of the total surface area of particles comprising polar stratospheric clouds, for example, suspected as catalysts for ozone decomposition, can be potentially important to the study of the rate of ozone layer depletion.

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PROGRESS DURING FY 1994:

This work has resulted in an experimental study of diffusional coarsening, or Ostwald ripening, in a liquid-liquid two-phase system utilizing a 100 micrometer pathlength test cell. The discrete phase was nucleated on one wall of the cell which resembles island formation in thin film growth. Observation of Ostwald ripening over a period of  $1 \times 10^7$  s (~ 4mo.) reveals that droplet number decay and the average radius increase, in the asymptotic limit, is in good agreement with theoretical predictions for diffusional growth of spherical caps on a two-dimensional substrate which is a valid approximation for the geometry of this experiment. This kind of geometric configuration is often encountered in island evolution dynamics and phase segregation in thin films. Two approximate solutions were developed, one to monopolar order, and the other to the dipolar order. Both approximations closely follow the experimentally observed scaling laws characteristic for the mixed-dimensional coarsening. Certain deviations appearing among the two approximate solutions and the experimental data, however, are suggestive of differences between global and local effects.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-26-08-13

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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### *Electronic Materials*

PRINCIPAL INVESTIGATOR: Dr. Thomas K. Glasgow

NASA Lewis Research Center (LeRC)

#### CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

Particular attention is given to transparent materials of technical interest as modulators and detectors and of scientific interest as demonstration materials for pattern formation and fluid flow.

#### TASK DESCRIPTION:

The phenomena being studied include diffusion, coarsening, solution crystal growth, physical vapor transport, pattern formation in solidification of aggregation, solute rejection and transport, and nucleation behavior. The gravitational acceleration considered ranges from constant 1-g to low- and variable g-levels (g-jitter).

#### TASK SIGNIFICANCE:

The important feature of this work is the coordinated approach; i.e., quantitative agreement is sought between physical and numerical experiments. Attention must therefore be given to the development of diagnostic tools as well as to advanced numerical techniques.

#### PROGRESS DURING FY 1994:

Measurements of relative solid and liquid thermal conductivity have been made in support of ground-based research. Work has continued on algorithms describing pattern formation and on mixing in variable acceleration environments.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 10/92 EXPIRATION: N/A

PROJECT IDENTIFICATION: 962-21-05-02

RESPONSIBLE CENTER: LeRC

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*Combustion Synthesis of Materials in Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Irvin Glassman

Princeton University

## CO-INVESTIGATORS:

Dr. Kenneth Brezinsky

Princeton University

Prof. C.K. Law

Princeton University

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**TASK OBJECTIVE:**

The experimental investigation involves detailed probing and quantification of the heterogeneous flame structure, and the materials characterization of the nitride powders obtained. It builds on the experience already gained in a related NSF-supported program examining the gas phases combustion synthesis of materials through metal/halide exchange reactions. The theoretical investigation involves analyses of the propagation, structure and stability of the heterogeneous flame, and the reaction mechanisms of individual metal particles in variable density.

**TASK DESCRIPTION:**

A comprehensive experimental and theoretical program has been initiated to synthesize metallic and nonmetallic nitrides (especially titanium nitride) under microgravity conditions, and to understand the underlying combustion mechanisms. The progress applies the Self-propagating, High-temperature Synthesis (SHS) technique to titanium (or other metal) particle suspensions in supercritical nitrogen: the nitride particles are formed upon passage of a self-sustained flame through this suspension.

**TASK SIGNIFICANCE:**

Microgravity prevents particle settling, agglomeration, and liquid/metal film formations and permits the use of suspensions of particles of specified characteristics. This facilitates the production of nitride powders of high purity, uniformity, and specificity. The novel use of supercritical nitrogen to provide a supply of reactant at conditions near the critical temperature and pressure avoids bubble formation that occurs when liquid nitrogen is used and also provides a means for greatly changing reactant density with a modest change in pressure because of the very high isothermal compressibility near the critical point.

**PROGRESS DURING FY 1994:**

Titanium powders immersed in near supercritical nitrogen in a temperature range of 126.26-141.75 °K and a pressure range of 3.4-6.8 MPa, have been converted to titanium nitride by self-propagating high-temperature synthesis (SHS). Both packed pellets and loose powders have been nitrided by this method. The conversion yields to titanium nitride for the loose powders are higher than those reported in the literature for pure titanium powders combusted in nitrogen at any other conditions. However, the conversion yields for the packed titanium powders were significantly lower than those for the loose powders and lower than obtainable when liquid nitrogen instead of supercritical nitrogen was used. In addition to the titanium powders, loose tantalum powders have been nitrided in supercritical nitrogen but the yields obtained are lower than those reported in the literature for room temperature ignition at comparable pressures. Loose silicon powders could not be ignited in this media.

In order to conduct the experiments described above, a cryogenic, constant volume reactor was designed and constructed. The reactor not only met the materials requirements to safely contain a high pressure, low temperature, supercritical nitrogen medium, but also generated the nitrogen working conditions from standard nitrogen sources. The reactor was equipped with optical access ports so that the progress of the combustion synthesis could be filmed.

Progress in the theoretical understanding of nonadiabatic heterogeneous flame propagation and extinction was achieved through an analysis based on a premixed mode of propagation for the bulk flame supported by the non-premixed reaction of dispersed non-metals in the liquid metal. The formulation allows for volumetric heat loss



throughout the bulk flame, finite rate Arrhenius reaction at the particle surface, and temperature sensitive Arrhenius mass diffusion in the liquid.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 11/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 962-26-05-09

NASA CONTRACT No.:NAG3-1418

RESPONSIBLE CENTER: LeRC

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*Evolution of Microstructural Distance Distributions in Normal Gravity and Microgravity*

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PRINCIPAL INVESTIGATOR: Prof. Arun M. Gokhale

Georgia Institute of Technology

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

1. To develop the methodology for estimation of distributions of distances between microstructural features in microstructures by using digital image analysis and stereological techniques.
2. To apply the methodology to study the evolution of microstructural distance distributions during some important materials processes such as: particle coarsening, liquid phase sintering, etc.
3. To quantify the relative contributions of intrinsic process dynamics and gravity on the evolution of microstructural spatial distance distributions.

**TASK DESCRIPTION:**

Digital image analysis techniques will be applied to obtain the centroid coordinates and the size of each microstructural feature of interest observed in a two-dimensional section through three-dimensional microstructure. The 2D microstructural distance distributions will be extracted from these data via a suitable computer code. Stereological methods will be utilized to estimate the true 3D spatial distance distributions from the apparent 2D distance distributions. The methodology will be applied to study the evolution of distance distributions during particle coarsening and liquid phase sintering in normal gravity, and subsequently in microgravity.

**TASK SIGNIFICANCE:**

An important benefit of processing of materials in microgravity would be the control of distribution of spatial distances between microstructural features, and the consequent gain in the material properties. This research program will develop the techniques for quantitative characterization of spatial distance distributions to objectively gauge the relative contributions from intrinsic materials process dynamics and gravity to the evolution microstructural distance distributions. The resulting information should be very useful to understand and model the role of gravity on microstructural evolution during materials processes.

**PROGRESS DURING FY 1994:**

This research project started on July 11, 1994. During the first three months, the efforts were devoted to complete the development of the interactive software to extract the centroid coordinates of microstructural features in a series of microstructural fields (say 50 contiguous fields) referred to the same origin, so that the distances between the features, not necessarily in the same field of view, can be computed. The technique will be applied to obtain some preliminary data on some specimens of liquid phase sintered material provided by Professor R.M. German of Pennsylvania State University.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/94 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-25-05-29

NASA CONTRACT No.: NAG3-1651

RESPONSIBLE CENTER: LeRC

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*Influence of Free Convection in Dissolution*

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PRINCIPAL INVESTIGATOR: Prof. Prabhat K. Gupta

Ohio State University

CO-INVESTIGATORS:

S.A. Korpela

Ohio State University

A.R. Cooper

Case Western Reserve University

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**TASK OBJECTIVE:**

To study the influence of natural convection in dissolution of silica glass into a melt consisting of  $PbO$  and  $SiO_2$ .

**TASK DESCRIPTION:**

The following two-part task has been undertaken. The first portion of the task consists of designing an experimental apparatus. The second portion of the task involves the design of a computer program for theoretical calculations. Each portion of the task is under way.

**TASK SIGNIFICANCE:**

The aim of the research is to understand the influence of free convection in dissolution dynamics of solids subjected to a corrosive environment. In terrestrial applications a better understanding of this phenomenon should lead to savings by way of improved life of silica liners in containers used in batch processing of various corrosive materials. The aim of this task is to achieve a good enough theoretical model so that the proposed ground based experiments can be faithfully simulated via computer calculations.

**PROGRESS DURING FY 1994:**

The composition of 50 mole percent  $PbO$  - 50%  $SiO_2$  has been selected as the melt and pure silica as the solute to examine the influence of free convection on dissolution. The density variation with mole percent of silica is found to be linear with a slope of 0.081 g/cc per mole percent of  $PbO$ . A student in Mechanical Engineering has initiated setting up the fundamental equations describing the dissolution phenomenon in presence of free convection.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0

MS Students: 1

PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 5/95

PROJECT IDENTIFICATION: 962-25-05-24

NASA CONTRACT NO.: NAG3-1602

RESPONSIBLE CENTER: LeRC

*Noncontact Thermal, Physical Property Measurement of Multiphase Systems*

PRINCIPAL INVESTIGATOR: Dr. Robert H. Hauge

Rice University

CO-INVESTIGATORS:

J.L. Margrave

Rice University

**TASK OBJECTIVE:**

The long range goal this equipment development effort is to create a prototype measurement facility for the physical and chemical properties of levitated samples. Our objectives are as follows:

1. To determine the capabilities and accuracy of newly developed techniques for measurements of surface optical properties, density, temperature and surface tension of levitated solids and liquids.
2. To test the overall performance of current concentrators in combination with controlled gas flows and axial DC magnetic fields with respect to the creation of axially symmetric RF fields and control of liquid sample rotation, vibration and translation.

**TASK DESCRIPTION:**

Measurement techniques for the properties are as follows:

1. **Temperature:** A simultaneous measurement of sample brightness at a vareity of wavelengths through the visible and near infrared is obtained along with the sample emissivity and optical constants at 632 nm.
2. **Density:** A triggered high-resolution video camera image of the liquid drop is obtained with backlighting of the sample by a 670-nm diode laser. Images are taken when the sample has spherical symmetry, and the density is obtained from a calculation of volume with use of an accurately determined sample profile.
3. **Surface tension:** Surface-tension measurements are obtained from the vibrational frequencies of the oscillating drop. The vibrational frequencies are obtained from a frequency analysis of reflected or emitted light.
4. **Electromagnetic levitation coils:** Current concentrators which provide both vertical and lateral electromagnetic symmetry have been developed for efficient EM coupling to the sample and improved symmetry of the positioning forces. Symmetries other than cylindrical are under investigation as a means of controlling sample rotation.

**TASK SIGNIFICANCE:**

Accurate methods for measuring liquid metal physical properties will permit improved modeling of liquid metal flows in direct coating of metals to near final dimensions.

**PROGRESS DURING FY 1994:**

Measurements of the temperature dependence of the optical constants and emissivity of gold has demonstrated the accuracy of the PEM ellipsometry method for levitated samples.

Surface tension measurements of gold indicate the current methods of modelling the effects of external levitation forces do not work well for samples with large amplitude motions.

Coupling of large DC currents to the levitation coil was found to be not effective in suppressing sample rotation.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

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TASK INITIATION: 12/91    EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-25-07-04

RESPONSIBLE CENTER: JPL

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**Proceedings**

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*Microgravity Processing of Oxide Superconductors*

PRINCIPAL INVESTIGATOR: Dr. William Hofmeister

Vanderbilt University

## CO-INVESTIGATORS:

R. Bayuzick

Vanderbilt University

R. Hopkins

Westinghouse S&amp;TC

M. Vlasse

NASA Marshall Space Flight Center (MSFC)

## TASK OBJECTIVE:

Since the discovery of superconductivity in lanthanide-based perovskite systems, considerable effort has concentrated on the synthesis and characterization of these materials. Melt processing techniques have shown great promise in developing bulk crystalline materials with critical current densities necessary for practical application. The YBaCuO system has received the most intense study, as this material has shown promise for the application of both thin film and bulk materials. However, little information is available on the complete melting relations, undercooling, and solidification behavior of these materials. In general, the understanding of undercooling and solidification of high temperature oxide systems lags behind the science of these phenomena in metallic systems. Therefore, this research will investigate the fundamental melting relations, undercooling, and solidification behavior of oxide superconductors through melt processing, with an emphasis on improving ground based synthesis of these materials. The techniques developed by this project will apply to oxide superconductors in general and will provide additional avenues for research in this field. An additional goal of this program is to develop collaboration with other groups and provide access to the unique processing strategies developed by the microgravity program.

## TASK DESCRIPTION:

The proposed research will continue experiments with  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$  using drop tubes at Vanderbilt University, the 105 meter drop tube at Marshall Space Flight Center, and aero-acoustic levitation at Containerless Research, Inc. Microstructural and compositional analyses will be performed at both Vanderbilt University and the Westinghouse Science and Technology Center. Application of acoustic levitation and microwave heating at the Jet Propulsion Laboratory, electrostatic levitation at the Jet Propulsion Laboratory, undercooling combined with rapid heat removal in the Undercool and Rapid Quench Technology (URQT) drop tube at Vanderbilt, and addition of small amounts of Ag to the material for improving electromagnetic susceptibility will be explored and incorporated if the techniques prove useful. In order to correctly assess thermal profiles upon melting and solidification in aero-acoustic levitation experiments, the Ultra High Speed Thermal Imager (UHSTI) at Vanderbilt University will be utilized.

## TASK SIGNIFICANCE:

Since the discovery of high- $T_c$  superconductivity in copper oxide systems, much progress has been made in the area of thin film technology for small scale applications. However, the science suffers from the lack of crystalline materials with high critical current densities that are large enough for bulk or large scale applications. Melt processing in general has proven to be the best technique for producing bulk materials with significant critical current densities ( $<10^4$  amp  $\text{cm}^{-2}$  in melt texturing experiments).

The novel techniques developed in this study have brought new insight into the understanding of the phase relations in this complex system. These enhancements will help to advance the understanding of the science of high- $T_c$  superconductivity and of the material properties of oxide superconductors. Advancements have a direct impact on the production of bulk materials for large scale applications.

**PROGRESS DURING FY 1994:**

In addition to the drop tube experiments performed over the past three years, investigations have been initiated into aero-acoustic levitation (AAL) using the facility at Containerless Research, Inc. in Chicago, IL. This technique allows for processing samples ranging in size from 2 - 3 mm. In these experiments, samples were completely molten, and upon solidification, a pronounced recalescence event occurred. Two different microstructures resulted. One consisted of primary tetragonal  $\text{YBa}_2\text{Cu}_3\text{O}_{7.4}$  dendrites and a mixture of several phases including fine dendrites of  $\text{Y}_4\text{Ba}_3\text{O}_9$ . The second microstructure which developed consisted of single phase tetragonal  $\text{YBa}_2\text{Cu}_3\text{O}_{7.4}$ . The formation of a single phase indicates that samples were undercooled to well below 1273 K. Upon annealing in flowing oxygen, these samples revert to the superconducting orthorhombic structure and exhibit critical current densities on the order of  $10^3 \text{ A cm}^{-2}$  up to about 45 kOe applied magnetic field at 5 K.

Experiments have also begun to determine the electromagnetic susceptibility of  $\text{YBa}_2\text{Cu}_3\text{O}_{7.4}$  using RF frequencies in the megahertz range. These experiments will ultimately include small additions of Ag into the structure in an attempt to enhance electromagnetic susceptibility. Also, preliminary experiments have begun using the UHSTI during aero-acoustic levitation experiments.

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**STUDENTS FUNDED UNDER RESEARCH:****TASK INITIATION:** 11/94    **EXPIRATION:** 11/96**PROJECT IDENTIFICATION:** 962-25-08-22**NASA CONTRACT No.:** NAG8-832**RESPONSIBLE CENTER:** MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Journals**

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*Non-Equilibrium Phase Transformations*

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PRINCIPAL INVESTIGATOR: Dr. Kenneth A. JacksonUniversity of Arizona

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CO-INVESTIGATORS:

B. Zelinski

University of Arizona

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**TASK OBJECTIVE:**

Our computer simulations have resulted in a breakthrough in our fundamental understanding of the physics associated with phase changes which take place far from equilibrium. This includes solidification, especially rapid solidification, crystal growth, especially orientation effects on segregation, condensation from vapor, etc. The goal of this program is to continue computer simulations and to initiate an experimental study to explore the implications and insights provided by this breakthrough.

**TASK DESCRIPTION:**

The experiments involve an analysis of microsegregation that requires a microgravity environment to minimize the effects of convection. A program combining simulations with experimental studies is viewed as essential, not only in providing new insights for the experimental work, but also in keeping the simulations relevant to the real world. Experimental studies of the microsegregation of dye molecules during the crystallization of transparent organic compounds will be pursued. These experiments will require verification in a convection-free environment. The computer simulations to date have clearly demonstrated the difference between the diffusionless and the diffusion-dominated regimes of growth. There is still much to be done in this area, and the studies to date are but a beginning: they demonstrate that this method of simulation can indeed capture the essence of transformations that occur in the real world. This simulation method will be used to explore, in detail, the transition between the diffusionless regime and the diffusion-dominated regime. It will be used to predict growth rates, transformation temperatures, and transformation-induced segregation effects. It will be used to study the early stages of interface instability, including not only the growth rate dependence of the distribution coefficient, but also the effects of having a crystal composed of discrete atoms growing into an alloy that has randomly-distributed atoms rather than a crystal bounded by a mathematical surface growing into an alloy of uniform composition.

**TASK SIGNIFICANCE:**

The structure and properties of materials produced by transformations that occur under conditions that are far from equilibrium is one of the central topics of materials science. It has been observed experimentally in many materials that the distribution coefficient depends on the growth velocity and on the orientation of the crystal surface during growth. These are not equilibrium phenomena which can be described by the standard quasi-equilibrium thermodynamic model. Monte Carlo computer simulations have reproduced these phenomena and predict that they depend on the undercooling, on the growth rate, and on the diffusion coefficient in the fluid phase. These effects are particularly important in the rapid growth regime or more generally, in a regime where the phase transformation takes place far from equilibrium. Projects supported by NASA using levitation and the 0-g environment are making major contributions to our understanding of nucleation and crystallization under these conditions, in the regime where these non-equilibrium effects are very important. Our simulation results provide a new framework for the interpretation of these experiments.

**PROGRESS DURING FY 1994:**

An analytical model has been developed which describes most of the features of the simulations. The model predicts a growth rate curve which has a loop in it at high concentrations, so that there are regions where the growth rate is not a single-valued function of temperature. This loop accounts for oscillations in the growth rate which have been observed during laser melting of alloys, and for the jump in growth rate as a function of undercooling which has been observed in measured growth rates of alloy dendrites. The model includes the composition dependence of the



k-value discussed below. We have explored a range of compositions in the Monte Carlo modeling. The segregation coefficient in the simulations does not depend on composition for dilute alloys, but is strongly composition dependent for alloys with greater than about 5% of alloy concentration. These observations have led to the development of an analytical model based on a novel functional form for the k-value as a function of growth rate. Simulations in three dimensions show results which are similar to the simulations in two dimensions, but these are quite different from simulations in one dimension. We understand why this is so: the connectivity of the interface enables energy to be transferred between atoms at the interface. This can occur in two- and three-dimension, but not in one-dimension. Code is being developed to incorporate a temperature gradient into the Monte Carlo program for simulations of cellular growth. This code will also be capable of modelling the phenomena of fluctuating growth rates which have been observed during the laser melting of alloys. This phenomenon involves an interaction between the non-equilibrium growth rate and the temperature gradient. We have so far been unable to identify a system suitable for the study of the incorporation of dyes into organic crystals, although dozens of systems have been evaluated. We plan to invert this process to study the crystallization of organic dyes containing impurities. A Bridgman apparatus is being constructed which will be used to measure the growth rate and orientation dependence of the incorporation of dopants into InSb. A solution growth apparatus is planned which will be used to measure the growth rate and orientation dependence of impurity incorporation into water soluble crystals during growth. These ground-based experiments are subject to uncertainties due to convection, and the experimental results will require verification in a convection free, 0-g environment.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-25-08-29

NASA CONTRACT NO.: NAG8-944

RESPONSIBLE CENTER: MSFC

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Jackson, K.A. "Optical materials in microelectronics." Invited Presentation at the Glass Conference in honor of Prof. Norbert Kriedl, July, 1994.

*Combined Heat Transfer Analysis of Crystal Growth*

PRINCIPAL INVESTIGATOR: Dr. Mohammad Kassemi

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The technical objective of this research effort is to provide a quantitative understanding of the role of radiation heat transfer on crystal growth in low-gravity space experiments. To accomplish this goal a generalized combined conductive-convective-radiative numerical model is developed which can accommodate various crystal growth experiments.

**TASK DESCRIPTION:**

Radiation heat transfer affects crystal growth in both space and ground-based processing. This is due to the high operating temperatures of the crystal growth processes, the semi-transparency of the crucible and phase change materials and the heat transfer link between the crucible and the furnace. In ground-based experiments, radiation and convection coexist and compete for dominating the heat transfer process in the ampoule. The role of radiation, however, becomes more prominent in low-gravity environment of space where convection heat transfer is considerably minimized.

A numerical methodology was developed for radiation exchange in the generalized multi-dimensional cylindrical geometries encountered in crystal growth. The radiation scheme which is based on the Discretized Exchange Factor method (DEF) uses node-to-node exchange to calculate radiation heat transfer in an absorbing emitting scattering non gray medium. One of the advantages of this radiation model is that it can be easily incorporated into existing finite difference or finite element codes.

The radiation model was incorporated into a finite element code for fluid flow and heat transfer and a generalized radiation-convection-conduction model was developed for crystal growth in cylindrical ampoules. The solution algorithm tracks the movement of the interface during the solidification process and adjusts the finite element mesh to accommodate changes in the shape and position of the growing solid. Radiation view factors are also continuously update as the geometry is altered.

**TASK SIGNIFICANCE:**

Low gravity simulations performed using the combined heat transfer model, clearly indicate that radiation effects are dominant during solidification of oxide crystals, such as BSO, and may cause highly stretched and curved interfaces during low gravity solidification experiments. The convection-conduction models that do not include radiation effects rigorously cannot be used to predict the shape and movement of the solidification front correctly.

Thermal radiation governs the energy balance in many earth and space processes. It plays a crucial role in production of glasses, semi-conductors, and advanced pay-off materials such as optical crystals. This study is geared towards understanding the effects of radiation heat transfer on crystal growth in both low-g and 1-g environments and to seek possibilities of exploiting these effects for improving the quality of the growing crystal.

**PROGRESS DURING FY 1994:**

A generalized radiation-convection-conduction model was developed for crystal growth in Bridgman furnaces. The validity and accuracy of the radiation scheme was tested by comparing results to benchmark limiting solutions available in the literature. Combined heat transfer and fluid flow models were developed for solidification of two oxide crystals BSO (bismuth silicate) and YAG (yttrium aluminum oxide garnet). Both crystals are transparent to

radiation below 6 microns and opaque to radiation in the rest of the spectrum. The models are based on actual experimental configurations and conditions and include the effects of the furnace and the crucible wall. Results of numerical simulations which are also corroborated by qualitative experimental evidence clearly indicate the solidification of both BSO and YAG is greatly influenced by thermal radiation. The shape and position of the interface can be grossly misrepresented if radiation effects are neglected.

In low-gravity experiments, radiation loss through the semitransparent solid dominates the heat transfer at the solidification front for both materials and results in a highly stretched parabolic interface shape which protrudes convexly into the melt. As a result, the fluid flow pattern is also substantially modified.

In 1-g solidification of BSO, convection in the melt also plays an important role. In this case, heat transported by the recirculating vortices near the interface produces an interface which is much flatter than the corresponding low-g case.

Numerical experiments also indicate that because of the dominant role of radiation during solidification of semi-transparent crystals such as BSO and YAG, a flat interface shape can only be achieved by minimizing the net radiative loss from the interface through the solid. This is valid for both 1-g and low-g applications.

The study will be extended to include other oxide and semiconductor crystals with transparent bands. Effects of different crucible materials and furnace profiles will be investigated. A Monte Carlo radiation model will be developed to investigate the intricate effect of variation in the index of refraction across the interface.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-21-05-10

RESPONSIBLE CENTER: LeRC

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*Fundamentals of Thermomigration of Liquid Zones Through Solids*

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PRINCIPAL INVESTIGATOR: Prof. Michael J. Kaufman

University of Florida

## CO-INVESTIGATORS:

R. Abbaschian

University of Florida

A. Gokhale

University of Florida

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**TASK OBJECTIVE:**

Currently, significant resources in terms of expert manpower as well as direct costs are being expended to develop advanced materials such as the high-temperature structural intermetallics  $\text{MoSi}_2$  and  $\text{NiAl}$ , and the fast semiconductors  $\text{GaAs}$  and  $\text{GaSb}$ . Despite considerable efforts in these areas, the intrinsic properties of many of these compounds, and alloys based on them, remain poorly understood to the degree needed to enhance their development and application. For example, it is unclear if the brittle nature of many of the intermetallics of current interest is an intrinsic property of these compounds or is associated with interstitial impurities or impurity phases. Consequently, there is a need not only to produce single crystals of controlled purity, perfection and orientation, but also to understand the details of interfacial atomistics during crystal growth in order to bring about such control.

For many of the exotic compounds of technological interest currently, conventional crystal growth techniques have not proven successful in this regard because of the many problems associated with container contamination or loss of stoichiometry due to preferential vaporization of one or more of the elements with high vapor pressures (e.g. arsenic in  $\text{GaAs}$ , antimony in  $\text{GaSb}$ , silicon in  $\text{MoSi}_2$ , and aluminum in  $\text{NiAl}$ ). In addition, these conventional techniques are not amenable to a scientific study of correlations between thermosolutal convection and key aspects of crystal growth such as the interface (growth) temperature and morphological stability. For example, the use of electromagnetic induction to produce float zones introduces an additional electromagnetic stirring component in the liquid zone and also precludes the use of most sensors to measure interface temperatures.

**TASK DESCRIPTION:**

Although this method was discovered more than 35 years ago, there have been few attempts to utilize it as a means for processing materials in spite of the fact that most materials typically can be processed (e.g., grown as single crystals, joined and sectioned) at temperatures well below their melting points. In addition, coupling of this scheme with both acoustic emission/reflection and possibly Seebeck techniques should allow the interface positions, zone length, and interface temperature to be monitored *in situ* in real time.

**TASK SIGNIFICANCE:**

The focus of this research is intended to circumvent the common problems (e.g. crucible contamination and loss of stoichiometry) associated with conventional crystal growth methods by using the relatively unique technique of TGZM to grow single crystals of materials of current technological interest. In addition, the low-growth temperatures require much lower power inputs and result in enhanced safety during both ground operations and space flight. Also, it is anticipated that significant, hitherto unavailable data on liquid diffusivities will be generated during these experiments. Finally, it should be emphasized that the capability of producing high quality, inexpensive crystals of some of these rather exotic compounds could greatly facilitate advancements in these fields of current technological interest.

**PROGRESS DURING FY 1994:**

Research efforts in FY94 have focused on the design, assembly and implementation of experimental equipment to conduct the rather unique TGZM processing. Based on the considerable oxidation problems encountered in our initial studies in a relatively crude furnace with limited capabilities in terms of atmosphere control, a new TGZM furnace with high vacuum/purified inert gas capabilities was constructed and is currently being used for our attempts

at processing GaSb using a Ga zone. This vacuum chamber provides a sufficiently clean environment which has proven to be a critical factor in the development of this process in both the current GaSb/Ga and previous Si/Al systems. The current chill/furnace assembly produces temperature gradients across the zone comparable to those reported in early TGZM studies. Our preliminary attempts with the GaSb/Ga system have been encouraging and it appears that we are now in the position to concentrate on further development of the TGZM processing parameters, characterization of the critical factors related to the movement of the zone, and analysis of the various mechanisms involved in the TGZM process.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 2/93 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-21-08-16

NASA CONTRACT No.: NAG8-946

RESPONSIBLE CENTER: MSFC

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*Kinetics of Phase Transformations in Glass Forming*

PRINCIPAL INVESTIGATOR: Prof. Kenneth F. Kelton

Washington University, St. Louis

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this research are to develop computer models for realistic simulations of first-order phase transformations, in particular crystallization of liquids and glasses, and to design experiments to test those models. This research will lead to improved methods for the analysis of kinetic studies of these transformations, allowing, for example, kinetic parameters for nucleation and growth to be determined from peak profile studies of nonisothermal differential scanning calorimetry (DSC) measurements of crystallization. These new techniques will have wide applicability for phase transformation studies. In particular, they will allow real-time experiments of phase stability and transformation to be carried out in a microgravity environment.

**TASK DESCRIPTION:**

Glasses (primarily silicate based) that devitrify polymorphically by homogeneous nucleation are being studied experimentally and by computer modeling. Lithium disilicate ( $\text{Li}_2\text{O} \cdot 2\text{SiO}_2$ ) is used for most calculations and experimental measurements since the necessary kinetic and thermodynamic parameters are best known in that system. Other glasses studied include soda-lime silicate ( $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 3\text{SiO}_2$ ) and barium disilicate ( $\text{BaO} \cdot 2\text{SiO}_2$ ). The computer modeling of glasses (primarily silicate-based ones) is done at Washington University. The silicate glasses are prepared by Drs. C. Ray and D. Day of the University of Missouri, Rolla; they also study the devitrification kinetics experimentally with differential thermal analysis (DTA). Transmission electron microscopy (TEM) and DSC measurements are made at Washington University.

This research is a theoretical investigation of the nucleation and crystallization kinetics in glass forming systems. The importance of nonsteady-state nucleation and viscosity in glass formation is being investigated. Methods are being developed for the calculation of nonsteady state crystallization using isothermal and non-isothermal annealing. Calculations utilizing these methods on  $\text{Li}_2\text{O} \cdot \text{SiO}_2$  and  $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 3\text{SiO}_2$  will be performed, and several anomalous experimental results that appear to contradict the theoretical calculations will be investigated and explained.

**TASK SIGNIFICANCE:**

The computer models describing nucleation and growth under isothermal and non-isothermal conditions by simulating directly the evolution of the non-equilibrium cluster distribution will have wide applicability for phase transformation studies. In particular, they will allow real-time experiments of phase stability and transformation to be carried out in a microgravity environment.

**PROGRESS DURING FY 1994:**

During this past year, as part of the effort to make the computer modeling germane to phase transformations under realistic experimental conditions, the computer models were extended to model calorimetric data taken under isothermal and nonisothermal conditions in heterogeneously nucleating systems and to take into account effects due to finite particle sizes and surface crystallization. The experimental measurements were made in collaboration with Dr. C. S. Ray, University of Missouri, Rolla, MO.

1. Lithium disilicate glasses containing different, known amounts of Pt, a known nucleating agent, were prepared. Nucleation rates were measured directly and compared with theoretical predictions. The first detailed measurements of the transient time for heterogeneous nucleation were made and were found to agree with theoretical predictions made earlier by us. Experimental differential thermal analysis (DTA) measurements were made of the crystallization

of these glasses. An extension of our computer model tested earlier for homogeneously nucleating glasses was found to predict correctly changes in DTA peak parameters (peak position, width and height) with Pt concentration and DTA scan rate.

2. An effect of particle size on DTA peak parameters was established experimentally by us. SEM studies and DTA peak profiles indicated a considerable contribution of surface crystallization. Our computer programs were modified to take account of these effects by assuming random volume nucleation and growth and radial growth of a surface shell into the particle. Simulations were shown to agree qualitatively with the experimental data, though significant differences were noticed when the number of nuclei per particle became small. Ising-type lattice calculations demonstrated that our analytical expressions gave better account of finite size effects than the standard Johnson Mehl Avrami treatment, but failed when the number of nuclei became less than about five per particle (here represented by the lattice). An approximate analytical solution was developed for all numbers of nuclei per particle. Much better agreement was obtained with experimental data when this expression was incorporated into the computer simulation.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 2

TASK INITIATION: 5/91 EXPIRATION: 5/94

PROJECT IDENTIFICATION: 962-26-08-07

NASA CONTRACT No.: NAG8-873

RESPONSIBLE CENTER: MSFC

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Kelton, K.F. Numerical model for isothermal and nonisothermal crystallization of liquids and glasses. J. Non-Cryst. Solids, vol. 163, 283-296 (1993).

**Presentations**

Kelton, K.F. "Kinetics of phase transformations in glass forming systems." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

*Compositional Dependence of Phase Formation and Stability*

PRINCIPAL INVESTIGATOR: Prof. Kenneth F. Kelton

Washington University

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

## TASK OBJECTIVE:

The objectives of this research are to extend computer models developed by us to model first order phase transformations involving cases where the composition of the final phases are different from those of the initial ones. This study is important to resolve conflicts in the experimental literature, to develop theoretical methods for treating both steady state and time dependent nucleation in a partitioning system, to extend the understanding of basic nucleation phenomenon, and to aid in the eventual development of techniques for making qualitative predictions of desired phases and microstructures. This research will lead to improved methods for analyzing thermoanalytic data that will have wide applicability for phase transformations studies. In particular, it will aid in the design and data analysis of remote experiments, such as might take place in a microgravity environment. Drop-tube experiments may reveal new effects of composition on the nucleation of related complex periodic and quasiperiodic phases that will likely suggest future containerless solidification and microgravity studies.

## TASK DESCRIPTION:

The steady state and time-dependent nucleation rates as a function of temperature and  $\text{SiO}_2$  concentration will be measured in one pseudo-binary silicate glass,  $\text{Na}_2\text{O} \cdot \text{CaO} \cdot 3\text{SiO}_2$ . Initial work by others suggests a strong compositional dependence of the steady state nucleation rate, though the dependence of the transient behavior has not been measured. The concentration dependence of the maximum undercooling in drop-tube experiments in Ti-Mn-Si and Al-Cu-Co-Si, and if time permits, Ti-Cr-Si and Ti-Ni-Zr-Si, all metallic liquids that form quasicrystals, will be measured. These data will be used to refine a model, developed previously by us, of the magnitude and time dependence of the nucleation rate as a function of composition. That model for nucleation will then be incorporated in our numerical model for phase transformations. Initial tests of that extended model will be made by comparing calculated and measured trends of DSC data for the devitrification of  $\text{Na}_2\text{O} \cdot \text{CaO} \cdot 3\text{SiO}_2$  glass as a function of  $\text{SiO}_2$  concentration.

## TASK SIGNIFICANCE:

This research is a theoretical and experimental investigation of the nucleation and crystallization kinetics in silicate glasses and metallic alloys. The importance of partitioning on non-steady-state nucleation is being investigated. In addition to enhancing the basic understanding of nucleation in such cases, new methods, based on computer simulations, will be developed to analyze crystallization data under isothermal and nonisothermal conditions.

## PROGRESS DURING FY 1994:

Funding for this project commenced in August, 1994. Since that time, samples of Ti-Cr-Si and Ti-Cr-Si-O have been prepared and sent to Marshall Space Flight Center for drop-tube experiments. Samples of  $\text{Na}_2\text{O} \cdot \text{CaO} \cdot 3\text{SiO}_2$  glass are currently being prepared.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-26-08-14

NASA CONTRACT No.: NCC8-049

RESPONSIBLE CENTER: MSFC



*Solutocapillary Convection Effects on Polymeric Membrane Morphology*

PRINCIPAL INVESTIGATOR: Prof. William B. Krantz

University of Colorado, Boulder

## CO-INVESTIGATORS:

P. Todd  
R. OwenUniversity of Colorado  
Owen Research

## TASK OBJECTIVE:

- Design experiments for casting polymeric membranes under low-g conditions to study the influence of solutocapillary convection effects on membrane structure.
- Utilize the low-g environment provided by the KC-135 aircraft in order to conduct experiments to discriminate between two hypotheses which have been advanced to explain macrovoid-pore formation in polymeric membranes.
- Modify and couple an exiting model developed for the dry-cast membrane formation process to a model for macrovoid growth caused by solutocapillary convection.

## TASK DESCRIPTION:

In order to study the influence of solutocapillary convection on polymeric membrane morphology and to discriminate between the two hypotheses advanced to explain the growth of macrovoids in the dry- and wet-cast membrane-formation processes, the following tasks are identified:

1. Specification of membrane-casting solutions
2. Design and construction of the test cells
3. Perform ground-based control experiments
4. Carry out low-g experiments in KC-135 flights
5. Carry out structural characterization of membranes
6. Model development

## TASK SIGNIFICANCE:

Polymeric membranes are used for a variety of purposes ranging from water purification to environmental cleanup of effluent streams. Membranes remove impurities because of submicroscopic pores. One problem in creating submicroscopic pores in membranes is the propensity to form large pores or macrovoids which "short-circuit" the membrane's ability to separate dissolved impurities. Optimal means for eliminating macrovoids have yet to be developed owing to our lack of knowledge of what causes them. This research exploits the unique low-g environment to discriminate between two hypotheses advanced to explain macrovoid formation. The results of this research should enhance our ability to eliminate these undesirable defects during membrane manufacture.

## PROGRESS DURING FY 1994:

The following progress has been made in addressing the tasks listed above:

1. Specification of the membrane-casting solution: The composition range of the casting solutions has been determined within which macrovoid formation should occur. This has been established via model-based experiments. A novel technique was developed to stabilize these casting solutions against premature phase separation during the relatively long storage period prior to low-g flight experiments.

2. Design and construction of the test cells: A laboratory version of the test cell which closely resembles the flight-test version has been built and tested in ground-based experiments to insure that it can reproducibly cast ultrathin polymeric membranes under conditions in which macrovoids are observed. We also have successfully adapted this cell with an optical probe which will permit us to observe membrane formation in real time during the low-g experiments. This real-time measurement capability for studying membrane formation in low-g is novel and represents an added feature of these experiments which we did not anticipate in our original proposal to NASA. This feature will make these experiments of even more value than originally anticipated. Construction of the flight version of the test cell has begun.

3. Perform ground-based control experiments: We have chosen the cellulose acetate/acetone/water system as the casting solution from which our membranes will be formed. The test cell described above has been used to cast uniform reproducibly uniform-thickness membranes from these casting solutions in ground-based experiments. The resulting membrane structure has been studied using scanning electron microscopy (SEM), and macrovoids have been observed.

4. Carry out low-g experiments: The low-g flight experiments planned for the second half of year 1 have been booked for February of 1995, which is the tenth month of this project. Dr. Owen has provided consultation on the design of the flight experiments. Mr. Konagurthu, a chemical engineering student studying for his M.S. degree, is becoming qualified for flight via arrangements made through Marshall Space Flight Center.

5. Carry out structural characterization of membranes: Approximately 30 to 40 membranes cast via our ground-based experiments have been studied via SEM. The digital images from these SEM studies have been stored for comparison to those obtained from the membranes cast in low-g and the corresponding controls.

6. Model development: A coupled heat- and mass-transfer model for the evaporative casting of cellulose acetate membranes has been modified to properly describe the casting conditions of the special test cell we have designed and constructed for these experiments. This model has been used to determine the composition range of the casting solutions within which macrovoid formation might be anticipated. This model also has been used to design the capacity of the individual test cells in order to insure that the membranes can be completely formed during the 25 or so seconds of low-g time available in the flight experiments.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-26-08-15

NASA CONTRACT NO.: NAG8-1062

RESPONSIBLE CENTER: MSFC

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*Containerless Property Measurement of High-Temperature Liquids*

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PRINCIPAL INVESTIGATOR: Dr. Shankar Krishnan

Containerless Research, Inc.

CO-INVESTIGATORS:

P.C. Nordine

Containerless Research, Inc.

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**TASK OBJECTIVE:**

The task objective is to measure the optical properties of high-purity liquid metals and alloys (Al, Zr, Ni, and Ni-Zr alloys) over wide wavelength (220–1100 nm), temperature ( $T_m + 300$  K), and composition ranges under containerless conditions. Spectroscopic, pulsed dye-laser ellipsometry is used to obtain the complex dielectric functions and spectral emissivity data at high temperatures on clean liquids using electromagnetic levitation. These data are needed for accurate noncontact temperature measurement, and for measurements of the total hemispherical emittance by integration of emissivity over the wavelength range of thermal emission.

Another major outcome of this research will be the determination of the optical properties of liquids over a wide wavelength range. These measurements are of fundamental importance to advancing the theory of liquid metals. For example the presence or absence of important interband transitions provides information on the valence, bonding, joint density-of-states, and the extent to which nearly-free electron behavior is exhibited by liquid metals and alloys.

**TASK DESCRIPTION:**

The research approach is to levitate liquid metals and alloys (Al, Ni, Zr, and Ni-Zr alloys) electromagnetically and use a spectroscopic pulsed dye-laser ellipsometer to measure the complex dielectric function and the spectral emissivity in the wavelength range 220–1100 nm. Pulsed radiation is generated by a Molelectron dye-laser, and the wavelength is automatically set and adjusted by a laboratory computer. Light reflected by the specimen is collected and analyzed by a unique rotating analyzer ellipsometer.

The rotating components of the ellipsometer are motorized and controlled by the computer. The signals are detected by a pair of photodiodes and an EG&G boxcar averager is used to extract the mean value of the light intensities at the two photodiodes which receive the orthogonally polarized components of the reflected light. The signals are automatically measured by the computer.

A Molelectron dye laser is used to generate pulsed radiation in the 220–1100 nm wavelength range. The light is steered through several mirrors, and transmitted through a pair of Glan Thomson linear polarizers. The second polarizer is fixed in azimuth, and rotation of the first polarizer allows light levels to be adjusted to the optimum values. The light is incident on the liquid specimens at a fixed incident polarization, and the reflected polarization is analyzed for its new azimuth and ellipticity.

A modified rotating analyzer ellipsometer is used to measure the outgoing polarization at 6 azimuths of the analyzer. The analyzer is of the beamsplitting type such that the two orthogonal components of the beam are simultaneously obtained. Three intensity ratios are measured at three independent pairs of azimuths. The complex dielectric constant, indices of refraction, and spectral emissivities are derived from standard equations. The light intensities are detected by a pair of high speed, UV-enhanced silicon photodiodes and measured by a pair of gated integrators.

The emphasis of measurements on liquid metals and alloys is to determine the temperature and (for alloys) composition dependence of all optical properties over the accessible wavelength range. Measurements are possible from approximately 0.8 of the melting temperature, ( $T_m$ ) in undercooled liquids to at least 300K above  $T_m$ . The observed effects of temperature and composition on optical properties and the wavelength dependence of these properties are interpreted in terms of liquid structure and bonding.

The liquid nickel, zirconium, and their alloys have been chosen to be investigated in this research because they display unique glass forming behavior, typify early and late transition metals, and because they are also of interest to other NASA investigators. Measurements at 633 nm on these metals is being conducted in collaboration with Professors Bayuzick of Vanderbilt University and Johnson of the California Institute of Technology.

#### TASK SIGNIFICANCE:

The relevance and significance to the microgravity program are twofold. First, the spectral emissivity and total hemispherical emissivity form the basis for new noncontact thermophysical property measurements in microgravity experimentation. For example, knowledge of total hemispherical emittance allows heat capacity and thermal diffusivity to be accurately determined from free-cooling and pulse-heating experiments. Spectral emissivity data allow true specimen temperatures to be determined using optical pyrometry. Second, spectral emissivity measurements on specific materials are needed by other NASA investigators in ground-based and microgravity experimentation.

#### PROGRESS DURING FY 1994:

Work in the past year included spectral emissivity and infrared radiometric measurements on materials investigated on the TEMPUS flight experiment flown on the IML-2 mission. It allows the PI's to convert infrared radiance data obtained in the flight experiments to true specimen temperatures. The results of the work are summarized in a report to the TEMPUS PI's and to NASA. The conclusion of the report addresses the key issue of the need to cross-calibrate the laboratory and flight infrared radiometers. Following is the title and executive summary of this report. Part of the work has overlapped into FY 1995 funding period.

#### **Spectral Emissivities and Temperature Measurements: Liquid Zr, Ni, Ni-25% Sn, Ni-32.5% Sn, Ni-40% Nb, and Ni-75% Zr**

This work was conducted in support of the PI requirements for the TEMPUS flight experiment on IML-2. This report provides: (i) values of the spectral emissivities at  $\lambda = 633$  nm for liquid Zr, Ni, Ni-25%Sn, Ni-32.5%Sn, Ni-40%Nb, and Ni-75%Zr over a wide temperature range including undercooled and superheated conditions, (ii) experimentally determined spectral emissivity functions at  $\lambda = 633$  nm of the radiance temperature measured at  $\lambda = 650$  nm, which allow conversion of TEMPUS flight data to true temperature using the silicon-based flight pyrometer (SIP), (iii) experimentally determined functions that allow conversion of infrared radiance (1-2.5  $\mu$ m bandpass, ZPY-channel 1 on TEMPUS) data to true temperature, (iv) assessments of the total temperature errors, and (v) an analysis of the temperature measurement system. Pulsed-dye laser ellipsometry was conducted at  $\lambda = 633$  nm on electromagnetically levitated liquid metals and the radiance temperatures of the specimens were recorded using three pyrometers. Two of these pyrometers had an operating wavelength near 650 nm. The third pyrometer (LM) was developed by Heimann Optoelectronics GMBH and had a nominal operating wavelength range of 1-2.5  $\mu$ m. This pyrometer was constructed to be similar to the flight model (FM) and developmental model (DM), but exhibited significant differences from these pyrometers in its spectral sensitivity function. Most of the materials studied exhibited moderate temperature dependence of the optical properties and emissivities. Only nickel and zirconium could be undercooled significantly in our apparatus. Cross calibration of the LM and DM pyrometers will be needed, using the specimens of interest as the radiation source in order to interpret temperature measurements on TEMPUS in a manner that is traceable to a primary temperature standard. The infrared and 0.633  $\mu$ m emissivities of the materials were consistent with the behavior of a typical metal such as tungsten. A sensitivity analysis is presented using the actual wavelength dependence of emissivity for tungsten (over the bandpass of the LM and DM pyrometers) which showed that significant errors will remain if the present LM results were applied to correct apparent temperature measurements obtained on TEMPUS for emissivity effects. A direct comparison of the LM and DM pyrometers using a special tungsten strip lamp is recommended.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/92 EXPIRATION: 5/95

PROJECT IDENTIFICATION: 963-25-07-05

RESPONSIBLE CENTER: JPL

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### *Noise and Dynamical Pattern Selection in Solidification*

PRINCIPAL INVESTIGATOR: Prof. Douglas A. Kurtze

North Dakota State University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

To understand the fundamental mechanisms by which a pattern-forming system can change the spacing of its pattern, and to devise a means of calculating the preferred spacing to which external noise will drive the pattern.

#### TASK DESCRIPTION:

Numerical computation of the time evolution of mathematical models of solidification fronts, and analytical arguments to extract the preferred spacing from the model equations.

#### TASK SIGNIFICANCE:

Properties of materials grown from the melt are largely determined by their microstructure, which in turn is strongly affected by the front shape during solidification. This research will establish whether processing in a noisy environment (such as the Space Shuttle) can lead to more reproducible, and hence better controlled, front shapes than processing in a quieter environment. This study will definitively enhance our understanding of the fundamentals of solidification. A more complete study (such as this) is required for the design of materials with specific materials properties; a difficult task, but an important practical goal.

#### PROGRESS DURING FY 1994:

As of September, 1994, this grant has not yet been received. Nonetheless, it is possible, using older computer equipment, to run some of the smaller and simpler simulations involving perturbations of patterns governed by the Swift-Hohenberg equation and similar simplified, one-dimensional evolution equations.

An undergraduate research assistant, Jed Overmann, has been hired to work on this project. An incoming freshman, he is funded by the Science Bound program run ASEND, which administers the NSF EPSCoR grant to North Dakota. Before arriving, Jed had decided that his interests had changed and that he would prefer to work on some biological project. Thus he may not continue working on this project for the full academic year. However, he has continued working on it while seeking an appropriate and interesting project elsewhere, and his work has proven quite helpful.

I have written code to generate perturbed initial patterns for the Swift-Hohenberg equation and to integrate the evolution equation forward in time. Preliminary results indicate that perturbations to the amplitude of a cellular pattern are surprisingly ineffective at causing changes in the wave number of the pattern. Perturbations to the phase of the pattern are much more effective. For initial wave numbers which are well below the expected preferred wave number, we find that these perturbations do preferentially increase the wave number. We are currently working on quantifying and systematizing these results, and continuing to search through different initial wave numbers.

#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 10/94 EXPIRATION: 10/96

PROJECT IDENTIFICATION: 962-25-05-30

NASA CONTRACT NO.: NAG3-1603

RESPONSIBLE CENTER: LeRC

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Chemical Vapor Deposition of High  $T_c$  Superconducting Films in a Microgravity Environment

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PRINCIPAL INVESTIGATOR: Prof. Moises Levy

University of Wisconsin, Milwaukee

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CO-INVESTIGATORS:

B. Sarma

University of Wisconsin

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**TASK OBJECTIVE:**

The basic goal of this research is to grow and process single-crystal, high-temperature superconducting thin films under microgravity conditions.

**TASK DESCRIPTION:**

Superconducting thin films will be grown by chemical vapor deposition in three different transport reactors in order to investigate the effects of gravitationally-induced convection currents on the superconducting characteristics of the films. Different orientations with respect to gravity will be used.

**TASK SIGNIFICANCE:**

It is believed that microgravity will allow for unique growth conditions for vapor-phase transport and for the formation of single crystalline materials during postprocessing to the superconducting phase.

**PROGRESS DURING FY 1994:**

A compact chemical vapor deposition MOCVD system with a single organometallic precursor was designed, built and tested. A model for the vaporization and mass transport of mixed organometallics from a single source for thin film MOCVD was developed. It was shown that a stoichiometric gas phase can be obtained from a mixture of the organometallics in the desired mole ratios, in spite of differences in the volatilities of the individual precursor compounds. Proper composition and growth rate can be obtained by insuring that the organometallics are completely sublimed after the slotted borosilicate tube containing the compacted mixed organometallics has traversed the short heating zone of the vaporizer.  $Y_1Ba_2Cu_3O_7$  superconducting films with  $T_c$ 's around 90K were obtained on [100] Y stabilized cubic zirconia and [100] MgO substrates. Under ideal conditions the high  $T_c$  films were oriented with the c-axis perpendicular to the substrate plane. This is the preferred orientation for achieving large current densities in the plane of the film. The pressure of the argon carrier gas was between 5 and 7 torr and that of the oxygen gas was also within this range. The combined flow rate was between 200 and 400 standard cubic centimeter per minute.

The compact MOCVD system may be rotated in order to determine the effect of gravitational fields on the growth behavior of the high  $T_c$  superconducting films. In the first orientation investigated, the compacted precursor vaporizer tube was moved downward through the heating zone and the heated substrate was held upside down in the reactor arm of the U shaped system. Thus the gas flow rate past the reactor was against the gravitational field. It was found that the central part of the circular film was about 250 nm thinner than the outer part for a 1000 nm average thickness film. Thus fluid hydrodynamics affected the diffusion controlled growth rate of the films even at the low pressures of the MOCVD system. A microgravity environment may allow the use of lower gas flow velocities thus making the diffusion controlled growth rate process more uniform along the diameter of the films. Two other configurations of the MOCVD system with respect to the gravitational field are being investigated.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/91 EXPIRATION: 6/94

PROJECT IDENTIFICATION: 962-26-05-07

NASA CONTRACT NO.: NAG3-1274

RESPONSIBLE CENTER: LeRC

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*Microstructural Development during Directional Solidification of Peritectic Alloys*

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PRINCIPAL INVESTIGATOR: Dr. Thomas A. Lograsso

Iowa State University

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The mechanisms of and conditions required for the formation of peritectic solid dendrites, cells, or planar growth between the primary solid dendrites or cells and the transitions between these structures will be investigated. In addition, alternating bands of solid phases perpendicular to the growth direction have been observed in peritectic systems. However, the precise conditions under which these bands form and the mechanisms by which these bands form are not clearly understood. It has been postulated that the development of these different structures is related to changes in the composition of the liquid. A model will be developed that will predict the compositional variations that occur in the liquid under the assumption of no convection. This model will be compared to experimental results to evaluate the importance of convective effects on the formation of various structural characteristics.

**TASK DESCRIPTION:**

This research will focus on:

1. Establishing the growth conditions in terms of temperature gradient and growth rate for the formation of planar, cellular, and dendritic growth of the peritectic solid in a two-phase peritectic under convection-free conditions, with an emphasis on the morphological transition from cellular front primary and planar front peritectic solid to single phase planar front solid; and
2. Determining the effect of convection-free conditions on the formation of a banded structure during directional solidification of a two-phase peritectic alloy.

The research approach is to initially focus on two alloy systems, In-Sn and Sn-Cd. In-Sn has a very low peritectic temperature, which allows for the possibility of direct observation during solidification. In addition, In and Sn have essentially equal densities in the liquid state in the temperature range of interest, which should minimize density-driven convection. In the Sn-Cd system, the solute element (Cd) is more dense in the liquid state, which should minimize density-driven convection when solidification occurs upward. This system has been well characterized and is more tractable in terms of sample preparation than In-Sn. These results can be compared to previous work on the Pb-Bi system, in which the solute element (Bi) is less dense and thus provides a driving force for mixing in the liquid through convection.

**TASK SIGNIFICANCE:**

This research effort will establish the ground-based data required for the identification of the appropriate alloy system, composition, and growth conditions for microgravity processing. Studying solidification for these types of systems is important, because a number of industrially significant materials solidify in a similar manner such as brass, bronze, some steels and high temperature superconductors. The utilization of a microgravity environment greatly facilitates the analysis and modeling of data, since gravity-induced mixing in the liquid can strongly affect the solidification process.

**PROGRESS DURING FY 1994:**

The research effort has been concentrated in the following three areas.

1. The In-Sn phase diagram has been redetermined using thermal analysis in the region of the peritectic reaction. The composition range along the peritectic isotherm was found to be significantly more narrow than in currently published phase diagrams. Alloy compositions necessary to produce the microstructures of interest will be selected on the basis of this information.
2. Initial experiments have been conducted using Sn-Cd alloys to correlate microstructural development and interface morphology to growth conditions. The fraction solidified at the point of quenching will be varied to check the possible extent of convective effects.
3. A model has been developed to describe the formation of banded microstructures. Experimental results will be used to evaluate its predictions.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/93    EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-25-08-28

NASA CONTRACT No.: NAG8-963

RESPONSIBLE CENTER: MSFC

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*Numerical Investigation of Thermal Creep and Thermal Stress Effects in Microgravity Physical Vapor Transport*

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PRINCIPAL INVESTIGATOR: Dr. Daniel W. Mackowski

Auburn University

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CO-INVESTIGATORS:

R. Knight

Auburn University

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**TASK OBJECTIVE:**

The objective of this research is to quantitatively predict the roles of thermal creep and thermal stress on microgravity physical vapor transport through detailed numerical calculations.

**TASK DESCRIPTION:**

The research will formulate thermal creep and stress effects into a variable property, two-dimensional representation of heat and mass transfer in cylindrical ampoules under typical operating parameters. Physical conditions at which thermal creep and stress significantly alter vapor transport from a pure diffusion-limited mode, as well as the resulting effects upon mass transfer rates and mass flux uniformity at the crystal surface, will be determined. The research will benefit the prediction and development of microgravity crystal growth technology by providing, for the first time, an accurate accounting of all relevant physical vapor transport mechanisms.

The potential for growing high-quality crystals in closed, cylindrical ampoules using chemical vapor transport and physical vapor transport processes under microgravity conditions has been demonstrated in a number of experiments. The development of this technology will require an accurate modeling of the relevant vapor transport mechanisms occurring in buoyancy-free environments. Previous numerical efforts to predict microgravity crystal growth rates have employed a diffusion-limited formulation of vapor transport. However, theoretical arguments strongly indicate that the non-isothermal conditions encountered in ampoules can lead to an appreciable motion of the bulk fluid as a result of previously unrecognized phenomena. Temperature gradients tangential to the ampoule side walls can, through the mechanism of thermal creep, drive a slip flow of gas over the surface. In addition, temperature gradients within the gas itself can induce a fluid stress, and resulting fluid motion, through the mechanism of thermal stress. Order-of-magnitude estimates suggest that, under typical ampoule conditions, the fluid velocities resulting from thermal creep and thermal stress can be comparable to vapor diffusion velocities.

Specific tasks include:

- Develop code for numerically solving governing differential equations. Assemble thermodynamic and transport property database of commonly used PVT samples and carrier gases.
- Perform numerical calculations of diffusion-buoyant convection vapor transport in the absence of thermal creep and thermal stress. Check numerical results with previously published results.
- Incorporate thermal creep boundary conditions and thermal stress constitutive law into numerical code. Determine model parameter values at which thermal effects significantly alter vapor transport from diffusion and buoyant convection predictions.
- Determine the individual effects of thermal creep and thermal stress on vapor mass transport rates and the uniformity of mass flux at the crystal surface. Based on numerical results, develop rational expressions for estimating the relative contributions of the thermal vapor transport mechanisms to mass flux as a function of ampoule, sample, and carrier parameters.

**TASK SIGNIFICANCE:**

Under earthbound conditions, heat and mass transfer within nonisothermal physical vapor transport (PVT) crystal growth ampoules will often be dominated by buoyant convection. The rationale for performing PVT experiments in microgravity conditions is that the buoyant convective mechanisms will be suppressed, and crystal growth will

occur in a diffusion-limited mode. However, qualitative arguments indicate that the convective mechanisms of thermal creep and thermal stress, which are usually negligible in comparison to buoyant convection in earthbound environments, could significantly effect mass transfer rates in microgravity PVT systems. A quantitative understanding of the role of these mechanisms in microgravity PVT crystal growth processes requires detailed numerical modelling.

### PROGRESS DURING FY 1994:

Our efforts during this period focussed on the development of a code for modelling mass, momentum, and energy transfer in binary PVT systems. Unlike previous modelling efforts, the code is based on a transient formulation. Beginning with a specified initial equilibrium condition within the PVT ampoule, the boundary conditions at the nutrient source end are changed to prescribed operating conditions over a fixed length of time (which corresponds to raising the source temperature and nutrient partial pressure to their steady-state values). The evolution of the nutrient mass fraction, temperature, and velocity fields within the ampoule are determined by a semi-implicit time integration scheme for the energy, species, continuity, and momentum equations. We have found that the transient approach actually leads to a more efficient and reliable determination of the steady-state condition than can be obtained from direct solution of the steady-state condition.

We have used the model to further examine the effects of thermal creep on mass transfer rates and uniformity in binary PVT systems. We have specifically examined a system representing the Copper Phthalocyanine (CuPc) PVT experiments performed by Debe et al., for which the temperature drop across the ampoule had the relatively large value of  $DT = 673 - 343 = 330$  K over a length of 8.5 cm. The partial pressure of the nutrient (CuPc) in this experiment was also at a trace level compared to the background gas. In the absence of thermal creep, the predicted maximum flow velocity within the ampoule (resulting from Stefan flow) was on the order of 0.5 mm/s. Including thermal creep in the boundary conditions resulted in a maximum velocity of 15 cm/s. Although the differences in calculated velocities with and without creep are profound, we found that the net rate of mass transfer at the crystal was increased by less than 5% with the inclusion of creep. The uniformity of mass flux at the crystal, however, was significantly altered by creep. For the pure diffusion case the flux at the crystal was essentially uniform. Including creep, on the other hand, resulted in a 50% change in mass flux from the ampoule centerline to the side walls.

Further examination of thermal creep in  $\mu$ g PVT systems indicates that a critical factor is not necessarily the absolute change in temperature across the ampoule, but rather the local magnitude of the axial gradient of the wall temperature. Because the transition from the furnace 'hot' zone to the 'cold' zone can occur in a relatively small length, the axial temperature gradient in PVT systems can be locally high even in situations where the overall change in temperature across the ampoule is not appreciably large. Our predictions indicate that thermal creep can result in strong, locally-recirculating flows at the hot to cold transition point. If the transition point occurs in the vicinity of the crystal substrate, the creepinduced flows can appreciably alter the uniformity of growth.

We are currently modifying the code to include the effect of thermal stress, which arises from the contribution of the Burnett terms in the fluid stress tensor. We are also examining the effects of 'g-jitter' on the growth rates and patterns within the ampoule. Future work will focus on methods of allowing the crystal interface to become non-planar during the growth process, from which the stability of the interface can be deduced.

STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 6/93 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-21-08-18

NASA CONTRACT NO.: NAG8-977

RESPONSIBLE CENTER: MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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#### **Presentations**

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Walker, D.G., Mackowski, D.W., and Knight, R.W. "The effects of thermal creep on physical vapor transport under microgravity conditions." ALAA 32nd Aerospace Sciences Meeting, paper no. 94-0567, Reno, NV, January 10-13, 1994.

*Polymerizations in Microgravity: Traveling Fronts, Dispersions, Diffusion and Copolymerizations*

PRINCIPAL INVESTIGATOR: Prof. Lon J. Mathias

University of Southern Mississippi

CO-INVESTIGATORS:

R. Lockhead  
J. Pojman

University of Southern Mississippi  
University of Southern Mississippi

**TASK OBJECTIVE:**

Polymerization studies of bulk-phase diffusion and polymerization in monomer and comonomer mixtures or in monomer droplets in a non-solvent are being performed to study density effects on polymerization.

**TASK DESCRIPTION:**

These experiments will allow determination of fundamental monomer and polymer properties. The diffusion rate constants to be measured in the absence of convective mixing have not been accurately measurable on earth. This data will provide new insights into the fundamental behavior of polymers in a variety of situations important to their formation and processing. For example, molecular weights and distributions are directly dependent on the relative diffusion behavior of monomers and polymers. Processing of polymers, either in solution or from the melt, involves molecular reorganization and motion. The combination of convective mixing with diffusion determines the ability of polymers to effectively orient into crystalline domains. A further example is provided by copolymerizations at high conversion. In this regime, which is industrially important on Earth, the diffusion constants of monomers and polymers become extremely important. The copolymer composition throughout the reaction must be amenable to calculation and prediction. This is currently not possible without accurate knowledge of polymer and monomer diffusion constants.

Three separate but closely related research investigations are being conducted that all deal with developing a fundamental understanding of monomer and polymer diffusion during polymerization or processing under widely different conditions, and with applying this knowledge to the formation of unique materials. The three projects all involve bulk-phase diffusion and polymerization in neat monomer or comonomer mixtures, or in monomer droplets dispersed in nonsolvent. All three deal with diffusion effects that should uniquely manifest themselves under microgravity where analysis by optical, interference and light scattering will give complete understanding of macroscopic down to molecular level behavior.

**TASK SIGNIFICANCE:**

Microgravity offers a unique environment for studying polymer diffusion and polymer polymerization reactions. The absence of convection currents, which are the major mode of mixing at the molecular level on earth, are eliminated or reduced in the microgravity environment. More importantly, the prediction of unique copolymer composition development in microgravity will allow controlled formation of new compositions of matter. The absence of mixing at the molecular level should produce unique short-block copolymers available for the first time for comonomer compositions which normally lead to random or long-block copolymers under good mixing.

**PROGRESS DURING FY 1994:**

The opportunities for flying KC-135 and shuttle flight experiments are most exciting. They can immediately provide us with supporting results for the types of effects gravity are expected to have on several fundamental polymerization and phase separation processes. We will pursue possible KC-135 flight experiments with Jan Rogers on photoinitiated radical and cationic polymerizations of single and multicomponent materials. The former will serve as references and the latter will evaluate the effect of microgravity on phase separation during simultaneous polymerization of two miscible monomers that give immiscible polymers: kinetics of polymerization, demixing and aggregation. These experiments will be included in the shuttle experiments along with several additional systems testing spinodal decomposition of polymers dissolved in monomer solutions that undergo polymerization to give immiscible mixtures.

Our work on steric stabilization of oil/water systems has progressed with hydrophobically-modified hydrophilic polymers.

- Hydrophobically-modified poly(acrylic acid) is an effective steric stabilizer for oil-in-water emulsions, whereas hydrophobically-modified hydroxyethylcellulose seems to be incapable of stabilizing emulsion droplets below its critical overlap concentrations.

- Emulsion stability is obtained for both hydrophobically-modified polymers at concentrations that are vastly in excess of the critical overlap concentrations and that are postulated to be in the entanglement region.

We have made significant progress in our understanding of polymerization fronts. We have observed two instabilities in propagating fronts of methacrylic acid polymerization, both of which give the appearance of a spin mode. One occurs with liquid monomer when the tube is subjected to an external airflow to increase the rate of heat loss. It is a novel instability caused by convection related to bubble formation. Spinning modes very similar to ones observed in solid-state combustion reactions were observed when the initial methacrylic acid/initiator solution was maintained at 0 °C. The helical pattern left in the polymer and temperature profile measurements confirm for the first time the presence of a true spin mode in a constant velocity polymerization front. Using an IR camera, we were directly able to observe the spinning heads. Questions remain about the other spinning modes and the role of surface tension induced convection, which may be an excellent candidate for KC-135 flights.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 3

TASK INITIATION: 4/93 EXPIRATION: 4/95

PROJECT IDENTIFICATION: 962-26-08-10

NASA CONTRACT No.: NAG8-973

RESPONSIBLE CENTER: MSFC

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**Presentations**

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Khan, A., and Pojman, J.A. "Convective instabilities in propagating fronts of addition polymerization." Department of Chemistry and Biochemistry, University of Southern Mississippi, Mississippi Academy of Sciences, Biloxi. MS, February 17-18, 1994.

Lochhead, R.Y., and Rulison, C.J. "The adsorption of hydrophobically modified hydrophilic polymers to oil/water interfaces." American Chemical Society National Meeting, Washington, DC, August, 1994.

Lochhead, R.Y., and Rulison, C.J. "Adsorption of hydrophobically modified water soluble acrylic acid and cellulose based polymers at dynamic oil/water interfaces." 68th American Chemical Society Colloid and Surface Science Symposium, Stanford, CA, June, 1994.

Lochhead, R.Y., and Rulison, C.J. "The kinetics of polymer diffusion to liquid/liquid interfaces by drop volume techniques." Society of Cosmetic Chemists National Meeting, Las Vegas, NV, May, 1994.

Lochhead, R.Y., and Rulison, C.J. "An investigation of the ability of poly(acrylic acid) microgels to stabilize oil-in-water emulsion systems under shear flow conditions." Society of Cosmetic Chemists National Meeting, Las Vegas, NV, May, 1994.

Lochhead, R.Y., and Rulison, C.J. "A study of the adsorption of hydrophobically modified poly(acrylic acids) from aqueous solution onto hydrophobic/hydrophilic interfaces by densitometry and ultraviolet techniques." Society of Cosmetic Chemists National Meeting, Las Vegas, NV, May, 1994.

Lochhead, R.Y., and Rulison, C.J. "The kinetics of polymer diffusion to liquid/liquid interfaces by drop volume techniques." Southeastern Graduate Student Conference, Hattiesburg, MS, April, 1994.

Mathias, L.J. "Polymerizations in microgravity." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

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Mathias, L.J. "Convection instabilities in propagation fronts of addition polymerization." American Society of Mechanical Engineers, 14th Annual Winter Conference, New Orleans, LA, December 1, 1993.

Mathias, L.J. "Propagating fronts of polymerization." International Workshop on Dynamism and Regulation in Non-linear Chemical Systems, Tsukuba, Japan, March 23, 1994.

Mathias, L.J. "Factors affecting propagating fronts of polymerization." MacroAkron IUPAC International Symposium on Macromolecules, Akron, Ohio, July 11, 1994.

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*Quantitative Analysis of Crystal Defects by Triple Crystal X-Ray Diffraction*

PRINCIPAL INVESTIGATOR: Dr. Richard J. Matyi

University of Wisconsin, Madison

CO-INVESTIGATORS:

D. Gillies

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The objective of this task involves the development of various x-ray diffraction methods for the analysis of semiconductor materials, particularly ZnSe and its ternary alloys. The specific goals are (1) the development of high resolution triple crystal x-ray diffraction methods for the quantitative analysis of structural defects in semiconductor crystals; (2) the application of these analytical methods to compound semiconductor crystals (grown both in ground based experiments, and eventually, in microgravity) to achieve a better understanding of the effects of the crystal growth environment on the generation of defects; and (3) the transfer of this analysis technology to the Space Science Laboratory at the Marshall Space Flight Center (MSFC).

**TASK DESCRIPTION:**

The overall investigative approach of the research is the coupling of a new materials characterization tool (triple crystal x-ray diffraction) to the development of a new crystal growth system (ZnSe and its ternary alloys grown in microgravity). ZnSe and various alloy samples are being supplied by MSFC and are currently being analyzed by triple crystal diffraction methods to articulate the mosaic structure and the density of statistically distributed dislocations. Diffuse scattering is being used to monitor the surface preparation. Plans are being made to investigate the use of anomalous transmission as an alternate means of characterizing the defect structure, and to measure the volume fraction of twinned material using x-ray methods. The information obtained in these analyses is being forwarded to MSFC for corroboration by other analytical methods.

**TASK SIGNIFICANCE:**

The Microgravity Science and Applications Division at the Marshall Space Flight Center is engaged in a program under an approved flight experiment for the growth of the important semiconductor ZnSe and its alloys in space. Of prime importance in any crystal growth process is the reduction of the density of grown-in structural defects and the understanding of the relation between crystal growth and defect generation. Recent research at the University of Wisconsin - Madison has demonstrated that an analytical technique known as triple-crystal x-ray diffraction is capable of clearly differentiating the defect-free, "perfect crystal" regions of a sample from regions that are structurally defective. In this research program, we will use triple-crystal x-ray diffraction and related methods to examine the evolution of crystal growth defects as a function of crystal growth and relate the nature and quantity of these defects to the growth process. This differentiation and measurement of the effect of defects will be used to give a clearer insight into the effects of microgravity on the growth of technologically-important crystals.

**PROGRESS DURING FY 1994:**

Since this program began on July 1, 1994, we have been establishing the capabilities to perform the proposed research. Instrumentation has been modified to permit the analysis of as-grown ZnSe crystals, and preliminary analyses of samples have been performed. A graduate student has been hired to perform the research. A kick-off meeting has been scheduled for October 20, 1994 at MSFC to discuss preliminary results and to establish an analysis schedule.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/94    EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-21-08-18

NASA CONTRACT No.: NCC8-052

RESPONSIBLE CENTER: MSFC

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*The Interactive Dynamics of Convection, Flow and Directional Solidification*

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PRINCIPAL INVESTIGATOR: Prof. T. Maxworthy, Ph.D.

University of Southern California

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

In order to assess and control the effects of residual spacecraft accelerations on the growth of high quality crystals, full numerical simulations will be conducted. It is anticipated that this approach will lead not only to a minimization of the adverse effects of the flow on the crystal growth process, but also to the development of strategies for employing externally imposed flow fields to gain some degree of control over the solidification dynamics. Questions of interest concern the selection of length scales, the persistence of traveling waves, and of oscillatory instabilities in the nonlinear regime. Time dependent direct numerical simulations performed will allow investigation of these mechanisms in isolation, as well as with their combined effects.

**TASK DESCRIPTION:**

1. Study the types of morphological instability that are to be found during the directional solidification of binary mixtures;
2. Attempt the control of such instabilities using forced flow over the solidifying interface and assess the usefulness of reduced gravity in modifying the process, thereby improving the quality and reducing the defect in manufactured products.

**TASK SIGNIFICANCE:**

The basic mechanisms involved in controlling such nonlinear dynamical phenomena are of value to other fields of science and engineering, and the space environment may provide a suitable test bed for examining such ideas. This could be beneficial to control the solidification process by employing externally imposed flow fields to improve the quality and reduce defects in manufactured alloy products.

**PROGRESS DURING FY 1994:**

Since the award of this grant on June 16, 1994 we have concentrated our concerns on the design of the apparatus to carry out this task. During this period a Leitz Interferometric Microscope was made available by the Materials Research Section of NASA LeRC, and has been incorporated into the design. Some delays have resulted since the microscope was not physically available to us until the end of August. It now appears that our original concept will have to be modified to fit within the constraints imposed by the physical dimensions of the microscope. We anticipate the design and manufacture of the test cell will be completed by the end of November.

We have been fortunate in securing independent, one year funding, for a student to work on this project. He is a Chinese National from Northwestern Technical University in Xian, who has had considerable experience in this area of research. We anticipate significant progress once the apparatus is finally available for his use.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-25-05-26

RESPONSIBLE CENTER: LERC

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 $\text{Y}_2\text{BaCuO}_5$  Segregation in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  During Melt Texturing

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PRINCIPAL INVESTIGATOR: Dr. Paul J. McGinn

University of Notre Dame

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this ground based investigation is to understand the segregation of fine second phase  $\text{Y}_2\text{BaCuO}_5$  (211) precipitates during so-called "melt texture growth" of the superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_x$  (123).

**TASK DESCRIPTION:**

In melt texturing of 123, one of the goals is to refine the 211 that is present in the textured microstructure as much as possible to enhance flux pinning. However, segregation of fine 211 will affect the ability of the 123 to pin magnetic flux. It is unclear if segregation is an unavoidable consequence of precipitates being pushed by the solidification growth front, and what role is played by gravitational effects. The goal of this project is to understand the causes of 211 segregation. The effect of a number of factors on texturing will be examined to determine what is causing the observed segregation.

Through a series of melt texture growth experiments the effect of Pt additions on 211 size, and more importantly the 211 size dependence of segregation, will be determined. The effect of 123 growth morphology on 211 segregation will also be examined. This will allow for determination of whether the observed segregation is specific to only one morphology of 123 growth, or is characteristic of all 123 growth. The growth mode will be altered by using different techniques to achieve 211 refinement. It is anticipated that containerless processing experiments will be included in this part of the study as they will provide the best opportunity to observe impurity-free growth. A series of experiments will also be performed in which the viscosity of the melt will be altered to determine how the level of 211 segregation is affected.

After the effect of the above factors is understood experiments will be performed to attempt melt texturing of 123 in a microgravity environment.

**TASK SIGNIFICANCE:**

It is expected that this research will identify important factors affecting the segregation of fine 211 particles during the melt texturing of the 123-type high temperature superconductors. It will enhance the understanding of the crystal growth process and the development of the microstructure. It is anticipated that knowledge gained from working with this technologically important system will be applicable to other systems exhibiting similar crystal growth features.

**PROGRESS DURING FY 1994:**

A process for routinely growing Nd-123 seed crystals 2-3 mm on edge has been developed. These crystals are being used to seed the growth of Y-123 single crystals. Quenching from high temperature is being used to isolate growing Y-123 crystals. A mild etch process (acetic acid based) has been developed for extracting the crystals with only modest crystal degradation. Crystals are now being characterized as to the extent of 211 segregation and its relationship to the growth mechanism of the Y-123.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-21-08-29

NASA CONTRACT No.: NAG8-1064

RESPONSIBLE CENTER: MSFC

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*Interaction of Hele-Shaw Flows with Directional Solidification: Numerical Investigation of the Nonlinear Dynamical Interplay and Control Strategies*

PRINCIPAL INVESTIGATOR: Prof. Eckart H. Meiburg, Ph.D.

University of Southern California

CO-INVESTIGATORS:

T. Maxworthy

University of Southern California

**TASK OBJECTIVE:**

In order to assess and control the effects of residual spacecraft accelerations on the growth of high quality crystals, full numerical simulations will be conducted. It is anticipated that this approach will lead not only to a minimization of the adverse effects of the flow on the crystal growth process, but also to the development of strategies for employing externally imposed flow fields to gain some degree of control over the solidification dynamics. Questions of interest concern the selection of length scales, the persistence of traveling waves, and of oscillatory instabilities in the nonlinear regime. Time dependent direct numerical simulations performed will allow investigation of these mechanisms in isolation, as well as with their combined effects.

**TASK DESCRIPTION:**

1. Study the types of morphological instability that are to be found during the directional solidification of binary mixtures;
2. Attempt the control of such instabilities using forced flow over the solidifying interface and assess the usefulness of reduced gravity in modifying the process, thereby improving the quality and reducing the defect in manufactured products.

**TASK SIGNIFICANCE:**

The basic mechanisms involved in controlling such nonlinear dynamical phenomena are of value to other fields of science and engineering, and the space environment may provide a suitable test bed for examining such ideas. This could be beneficial to control the solidification process by employing externally imposed flow fields to improve the quality and reduce defects in manufactured alloy products.

**PROGRESS DURING FY 1994:**

Since the initiation of the grant on 6/16/94, design plans have begun for the apparatus to carry out the proposed tasks. We anticipate that the equipment will be in operation by February 1995. Efforts have been focused on the development and implementation of a highly accurate computational approach. Since the computational domain has a curved boundary at the solidification front, we employ a conformal mapping approach to obtain a rectangular domain. By assuming periodicity in the spanwise direction, the mapping can be performed analytically, which has the advantage of providing the analytical relationships between the original and the transformed variables. We have derived the relevant equations, boundary conditions and transformation terms in the computational domain. Since the problem is nonperiodic in the direction of solidification, we employ a compact finite difference method in that direction, while a spectral representation has been chosen for the spanwise direction. For the case without flow, we have numerically computed the growth rates of small wavy perturbations to the interface, and made preliminary comparisons with the classical linear stability theory of Mullins and Sekerka. The comparisons indicate excellent agreement.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0

MS Students: 0

PhD Students: 1

TASK INITIATION: 6/94 EXPIRATION: 6/96

PROJECT IDENTIFICATION: 962-25-05-27

NASA CONTRACT NO.: NAG3-1619

RESPONSIBLE CENTER: LeRC

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*The Synergistic Effect of Ceramic Materials Synthesis Using Vapor-Enhanced Reactive Sintering Under Microgravity Conditions*

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PRINCIPAL INVESTIGATOR: Prof. John J. Moore

Colorado School of Mines

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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## TASK OBJECTIVE:

The primary objective of this research is to develop a technique for processing advanced ceramics in the form of fine powders, whiskers or platelets. The secondary objective is to determine the extent of vapor phase reactions, including convection, in combination with gravity driven flow, on the process. Important concerns include product particle size and purity. Products should have particle sizes allowing for efficient handling and subsequent processing and/or function. Control of the chemical reactions and particle sized is necessary for the achievement of this objective.

## TASK DESCRIPTION:

Titanium diboride is synthesized according to the reaction  $\text{Ti} + 2\text{B} = \text{TiB}_2$  and titanium diboride + alumina ( $\text{Al}_2\text{O}_3$ ) composites are synthesized by the reaction  $3\text{TiO}_2 + 3\text{B}_2\text{O}_3 + 10\text{Al} = 3\text{TiB}_2 + 5\text{Al}_2\text{O}_3$ . The first reaction was chosen for its relatively simple solid-solid reaction, allowing the fundamentals of the reaction to be accurately evaluated. The  $\text{TiB}_2$  is formed via a combustion synthesis reaction in the propagating mode, which has high product purity and rapid reaction rates as inherent advantages of this process. The second reaction also uses the propagating mode, but is complicated by the fact that the reacting components become both molten and gaseous during the ignition process. Convection, therefore, plays a much greater role than in the first reaction. Both reactions will be carried out in both inert, i.e., Ar gas, and reactive, i.e., HCl gas, environments in order to evaluate the effects of vapor transport on the combustion synthesis reaction. A complete examination of the process variables, including micro and increased gravity, will be completed to examine the effect on the reaction.

## TASK SIGNIFICANCE:

Fine powders may be used as raw materials for subsequent processing techniques, while platelets and whiskers may be used as reinforcement materials in composite structures. Evaluation of reactions in microgravity enhance basic understanding of ground-based reaction mechanisms.

## PROGRESS DURING FY 1994:

Effects of gas pressure on the combustion synthesis of  $\text{TiB}_2$  in both argon and HCl have been investigated. In both cases as the pressure increases the ignition temperature increases and the combustion temperature decreases. It is also noted that as the combustion temperature increases the grain size of the product increases. Experiments in microgravity have been completed, and all reactions show a large increase in combustion temperature over ground-based results, as well as a more uniform grain size. Microgravity environments decrease the effect of convection, but the reaction mechanisms that depend on convection have yet to be fully clarified.

The alumina-titanium diboride system has also been investigated using reactive and inert gas pressure and microgravity. In this system, a vapor-liquid-solid reaction occurs at the reaction front, producing different results. Under normal conditions, gas pressure increases, ignition temperature decreases, and the combustion temperature increases, producing varied microstructures. In addition the reactive gas seems to have little effect on the combustion temperature. Though not yet fully analyzed, microgravity results in large increases in combustion temperature. However, microstructures still show large variations even within a given sample.



## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	1
PhD Students:	3	PhD Degrees:	0

TASK INITIATION: 1/93 EXPIRATION: 12/96

PROJECT IDENTIFICATION: 962-26-05-10

NASA CONTRACT No.: NCC-289

RESPONSIBLE CENTER: LeRC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Lantz, C., Moore, J.J., Readey, D.W., "Combustion synthesis of TiB<sub>2</sub> powders." ACerS 96th Annual Meeting, Indianapolis, IN, 1994.

Lantz, C., Moore, J.J., Readey, D.W., "Combustion synthesis of TiB<sub>2</sub> powders." AIChE National Meeting, Denver, CO, 1994.

Tefft, P., Moore, J.J., Readey, D.W., "Formation of Al<sub>2</sub>O<sub>3</sub>-TiB<sub>2</sub> composites with a high temperature synthesis reaction." ACerS 96th Annual Meeting, Indianapolis, IN, 1994.

Tefft, P., Moore, J.J., Readey, D.W., "Formation of Al<sub>2</sub>O<sub>3</sub> and TiB<sub>2</sub> powder mixtures by high temperature synthesis." AIChE National Meeting, Denver, CO, 1994.

### *Diffusion, Viscosity, and Crystal Growth in Microgravity*

PRINCIPAL INVESTIGATOR: Prof. Allan S. Myerson

Polytechnic University, New York

CO-INVESTIGATORS:

A. Izmailov

Polytechnic University, New York

#### **TASK OBJECTIVE:**

The results of this research should improve the operation and data analysis of existing microgravity crystal growth experiments and aid in the design of new hardware for such experiments.

#### **TASK DESCRIPTION:**

Components of this research include:

- Experimental studies of the diffusion coefficients and viscosity of triglycine sulfate (TGS), KDP, and other compounds of interest to microgravity crystal growth in supersaturated solutions as a function of solution concentrations, solution age, and solution history;
- Development of a theoretical model of diffusion and viscosity in the metastable state;
- Development of a model of crystal growth from solution including nonlinear time-dependent diffusion and viscosity effects;
- Employment of the model with and without buoyancy-driven convective flows to predict results of Earth and microgravity crystal growth experiments and to compare these results with experimental results; and
- Development of a computer simulation of the crystal growth process that will allow simulation of microgravity crystal growth, including the effects mentioned above.

Plans are to develop the adequate physical description of the crystal growth process in the supersaturated solutions. To achieve this goal, it will be necessary to take into account the nontrivial dependencies of such transport coefficients as diffusivity and viscosity on the solute concentration and solution temperature and age. The research program's approach will include obtaining the characteristic time for the crystal growth process in the supersaturated solutions, i.e., to obtain the duration of the metastable state relaxation that leads the system "crystal + supersaturated solution" to the equilibrium state. This equilibrium is the equilibrium between the crystal surface and the entire remaining volume of the surrounding saturated solution.

Another facet of the program will theoretically determine from the proposed model the difference in the concentration, temperature, and convective flow fields between an Earth-grown crystal and one grown in microgravity. This will also allow a comparison of the gains in face size and stability that might accompany microgravity crystal growth.

Comparison of theoretical predictions to the real experimental data will verify the theoretical model and determine numerical values for model parameters that have not been measured.

#### **TASK SIGNIFICANCE:**

Since crystallization from solution occurs in supersaturated solutions, the properties of these solutions and their role in nucleation and crystal growth are of interest. Recognition of this has led to a number of studies of supersaturated solutions and their properties.

**PROGRESS DURING FY 1994:**

Diffusion coefficients and viscosities of KDP, ADP and TGS have been measured over their entire undersaturated concentration range and in the supersaturated region.

An electrodynamic levitation technique was used to measure the water activity in KDP, ADP and TGS. This allows calculation of activity and activity coefficient for these materials in highly supersaturated solutions and allows estimation of the spinodal curve.

The diffusion boundary layer equations related to crystal growth from solutions are derived for the natural convection case with viscosity and density dependent on solute concentration. Solution of these equations has demonstrated that at the vicinity of the saturation concentration there is the following non-trivial dependence of the solution viscosity  $\eta$  on its density  $\rho$ :  $\eta = \eta(\rho) \rho^{1/2}$ . This theoretically obtained result has been verified by our experimental data.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 3/93 EXPIRATION: 3/96

PROJECT IDENTIFICATION: 962-26-08-09

NASA CONTRACT NO.: NAG8-960

RESPONSIBLE CENTER: MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

Myerson, A.S. "Diffusion, viscosity, and crystal growth in microgravity." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

Myerson, A.S. "New results for crystal growth from solution." Fifth Eastern Regional Conference on Crystal Growth, Atlantic City, NJ, October, 1994.

*An Electrochemical Method to Measure Diffusivity in Liquid Metals*

PRINCIPAL INVESTIGATOR: Prof. Ranga Narayanan

University of Florida

## CO-INVESTIGATORS:

T. Anderson

University of Florida

A. Fripp

NASA Langley Research Center (LaRC)

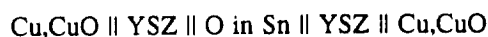
## TASK OBJECTIVE:

A research program will be conducted, that uses coulometric titration to measure oxygen diffusivity in liquid metals. Important aspects of the program are to estimate the effect of low gravity on the diffusivity measurements and to design experiments where the use of a low gravity environment significantly reduces the adverse effects of convection in the melt. The availability of these 'benchmark' values of diffusivity will be useful for assessing the reliability of different experimental designs and operational procedures for measuring diffusivity on earth.

Another science objective of this study is to establish a clearer picture of the constitutive behavior of 'Fickian diffusion' of oxygen in liquid metals. In particular, we will accurately measure the temperature dependence of the oxygen diffusion coefficient.

## TASK DESCRIPTION:

In this study an existing experimental 'cell' that has been modified by these investigators is used. An example of a system that is studied is the diffusion of oxygen in tin. The experimental arrangement consists of 2 electrochemical cells sharing a common working electrode, viz: tin. A representation of the cell is given by:



Here yttria stabilized zirconia (YSZ) is the solid state electrolyte through which oxygen ions are transported into or out of the liquid tin. In these experiments, one cell is used to establish a known boundary condition, usually a negligibly small concentration, while the second cell is operated in an open circuit mode to measure the concentration at the opposite boundary. The solid state electrolytes and tin are physically arranged so that the gravity vector is collinear with the gradient of concentration.

One of the YSZ plates is at the top and has a small overflow port to accommodate the expansion of tin, upon heating. Care is taken to avoid leakage of oxygen from this or other sources. Temperature gradients, in this so called isothermal experiment, are reduced in order to avoid natural convection under earth's gravity. Under earth's gravity, it is also important to ensure that oxygen concentration gradients are parallel to the gravity vector so that the melt is not 'top heavy'. This means that the oxygen concentration should be low at the 'bottom' of a vertically oriented cell. This in turn implies, that oxygen depletion takes place from the bottom or oxygen addition takes place from the top.

## TASK SIGNIFICANCE:

The availability of a method that gives benchmark values of diffusivity in liquid metals has many uses. These include the assessing of the reliability of different experimental designs and operational procedures for measuring diffusivities under Earth's gravity, as well as defining the constitutive relationships in liquid metals. This latter use is of fundamental importance in understanding the behavior of liquid metals and alloys.

**PROGRESS DURING FY 1994:**

The main result obtained thus far in the study may be summarized as follows:

The measured values of diffusivity depended on the cell size and experimental configuration. Measurements taken in configurations that gave rise to gravity driven convective flows also gave larger values of diffusion coefficients than measurements taken in configurations where the convective flows were apparently weaker. A detailed numerical model with a sophisticated 3-D code was developed, which predicts the effects of imperfections such as tilts and radial heat losses. Based on this modeling, it is concluded that even slight imperfections affect the diffusivity measurements for all of the cell configurations studied. Other observations that were influenced by the cell configurations were made. For example taller cells, which were associated with large time constants, increased the measurement error because of mass transfer through an overflow port. This port was included in the design in order to accommodate the expansion of tin upon heating. Thus, the initial oxygen concentration and cell dimensions both influenced the hydrostatic stability of the tin sample during a transient diffusion experiment. This is not surprising as the transport process is essentially the transient version of the Rayleigh-Bénard problem and the aspect ratio and Rayleigh number are two parameters which are found to define the stability criterion in that problem.

In brief, we believe that gravity driven convection in the melt prevents accurate measurement of the mass diffusivity of oxygen in metal systems by coulometric titration and thus likely in other measurement systems. This situation represents a clear example where a reduced gravity environment is necessary to obtain meaningful values of this important physical property.

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**STUDENTS FUNDED UNDER RESEARCH:**

BS Students:	2	BS Degrees:	2
MS Students:	1	MS Degrees:	1
PhD Students:	1	PhD Degrees:	0

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**TASK INITIATION:** 2/93 **EXPIRATION:** 2/96**PROJECT IDENTIFICATION:** 962-25-08-26**NASA CONTRACT No.:** NCC8-20**RESPONSIBLE CENTER:** MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

Narayanan, R., Anderson, T.J., and Fripp, A.L. "An electrochemical method to measure diffusivity in liquid metals."  
NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

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*Crystal Growth and Segregation Using the Submerged Heater Method*

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PRINCIPAL INVESTIGATOR: Prof. A. G. Ostrogorsky

Rensselaer Polytechnic Institute

## CO-INVESTIGATORS:

G. Müller

Universität Erlanger-Nürnberg

E. Monberg

AT&T Bell Labs

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**TASK OBJECTIVE:**

Fundamental studies of solute segregation at low levels of melt convection will be conducted using a programmable multi-zone furnace modified for growth by the Submerged Heater Method. The specific objectives of these studies are to:

1. Evaluate the suitability of the multi-zone furnace with the submerged heater to serve as a ground-based solidification facility, useful for pre-and post-flight studies.
2. Seek explanations for previous space experiments not demonstrating the diffusion controlled segregation.
3. Determine the criteria that will allow the diffusion-controlled segregation.
4. Evaluate the benefits of using the SHM within a PMZF for future space experiments.

**TASK DESCRIPTION:**

The research plan is to use the Submerged Heater Method in ground-based experiments for studies of mass transport in the melt at low Ra numbers. The effect of the equilibrium coefficient  $k$  will be particularly investigated. The studies will focus on electronics materials previously studied in space (e.g., Ge, InSb) doped with elements having different equilibrium distribution coefficients, ranging from  $k \sim 0.5$ , to  $k \sim 10^{-3}$ . Directional Solidification and zone melting procedures will be used during growth. The studies will be conducted using a programmable multi-zone Mellen furnace modified for the SHM. The ground-based experimental and theoretical work will be accompanied by numerical simulations. Numerical simulations will be used to model the transfer processes in the melt and to optimize the growth conditions. Future space experiments designed for the SHM will also be modeled.

**TASK SIGNIFICANCE:**

We hope to demonstrate that the multi-zone furnace with the submerged heater is useful for pre- and post-flight studies. The pre-flight studies may help to optimize the future space experiments. Our current "post-flight studies" focus on solvent-solute systems previously used in space (doped Ge, InSb and GaSb). We hope to explain why some space experiments did not result in diffusion controlled segregation.

**PROGRESS DURING FY 1994:**

Numerous modifications were made on the programmable 18-zone Mellen furnace. The furnace was calibrated and eight growth experiment with Ga-doped germanium were conducted. Convex solid-liquid interface and exceptionally uniform radial dopant distribution was obtained. We derived a nondimensional parameter, named the Segregation number that can be used to estimate the influence of convection on segregation. An axisymmetric model of heat and species transfer which allows studies of segregation in vertical Bridgman configuration (with and without the submerged heater) was developed.

A transparent two-zone Bridgman furnace was designed and built. The experiments in the transparent furnace are helping us to visualize melt convection and the shape and the position of the growth interface.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 4/93 EXPIRATION: 4/96

PROJECT IDENTIFICATION: 962-21-08-15

NASA CONTRACT NO.: NAG8-952

RESPONSIBLE CENTER: MSFC

## BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Ostrogorsky, A.G. "Crystal growth and segregation using the submerged heater method." Proceedings of the NASA Microgravity Materials Science Conference, Huntsville, AL (1994).

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Müller, G., and Ostrogorsky, A.G. "Convection in Melt Growth" chapter in "Handbook of Crystal Growth." North-Holland/Elsevier, vol. 2, pp 709-820, 1994.

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Ostrogorsky, A.G. "Directional solidification and zone melting using the submerged heater method." General Electric R&D Seminar, March 29, 1994.

Ostrogorsky, A.G., and Dagojlovic, Z. "Vertical Bridgman growth with and without a submerged baffle." 13th Conference on Crystal Growth, AACG/West, June, 1994.

Ostrogorsky, A.G., and Dagojlovic, Z. "Numerical simulation of heat and mass transfer in solidification by the submerged heater method." Gordon Research Conference on Crystal Growth, Proctor Academy, NH, June, 1994.

*Investigation of "Contactless" Crystal Growth by Physical Vapor Transport*

PRINCIPAL INVESTIGATOR: Dr. Witold Palosz

National Research Council (NRC)

CO-INVESTIGATORS:

Y. G. Sha

Universities Space Research Association (USRA)

D. Gillies

NASA Marshall Space Flight Center (MSFC)

C.H. Su

NASA Marshall Space Flight Center (MSFC)

S. Lowry

CFD Research, Inc.

TASK OBJECTIVE:

The primary objective of this study is to evaluate the conditions required for growth of crystals without contact with the side walls by physical vapor transport (PVT) in closed ampoules. The potentials and limitations of this technique ("contactless" PVT - cPVT) with respect to crystal size, growth rate, crystal quality, and the potential benefits of the microgravity environment for this technique will be assessed. The conditions for growth of Pb(Se, Te) by PVT will be determined. The evaluation of capabilities and limitations of the existing facilities and recommendations for space crystal growth facilities with respect to this PVT technique will be made.

TASK DESCRIPTION:

1. Development of numerical model of heat and mass transport in the growth system.
2. Development of thermochemical model of transport of Pb(Se, Te) by PVT.
3. Fabrication and /or modification of existing crystal growth furnaces.
4. Experimental investigation of transport of Pb(Se, Te) by PVT.
5. Performance of test crystal growth experiments.

TASK SIGNIFICANCE:

The benefits of the microgravity environment for crystal quality may be offset by strains and related crystal defects caused by the interaction of the crystal with the walls of the growth container. The cPVT technique offers improved growth conditions which may be beneficial for crystal growth both under ground and microgravity conditions. The study will provide a comprehensive assessment of the technique as opposed to the limited scope of the literature reports on the subject.

PROGRESS DURING FY 1994:

Budgeting arrangements are in process.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 2/95 EXPIRATION: 2/97

PROJECT IDENTIFICATION: 962-21-08-20

RESPONSIBLE CENTER: MSFC



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*Containerless Processing for Controlled Solidification Microstructures*

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PRINCIPAL INVESTIGATOR: Prof. John H. Perepezko

University of Wisconsin, Madison

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:****OBJECTIVE:**

The main research objective is the evaluation of the undercooling and resultant solidification microstructures in containerless processing, including drop-tube processing and levitation melting of selected alloys as an experience base for microgravity experiments.

**TASK DESCRIPTION:**

The degree of liquid undercooling attainable in a laboratory scale (3-m) drop-tube and levitation melting system can be altered through the variation of processing parameters such as melt superheat, sample-size, and gas environment. In a given sample, the competitive nucleation and growth kinetics between equilibrium and metastable phases controls microstructural development. The solidification behavior is evaluated through metallography, thermal analysis, and X-ray diffraction examination in conjunction with calorimetric measurements of falling droplets and a heat flow model of the processing conditions to judge the sample thermal history.

**TASK SIGNIFICANCE:**

In the current program studies, solidification microstructures are being examined in selected Ni and Mn based systems. The specific alloy selection is based on a metastable phase diagram analysis that allows for the identification of unique microstructures and microstructural transitions that may be produced by microgravity containerless processing.

**PROGRESS DURING FY 1994:**

A duplex partitionless solidification reaction involving fcc and bcc crystalline phases has been identified over a range of compositions near that of the eutectic in the Ni-V system. The reaction can be thought of as the limiting case of a eutectic transition ( $L \rightarrow \alpha + \beta$ ) in which  $\alpha$  and  $\beta$  have the same composition as the liquid phase. Drop tube experiments are being conducted to characterize the competitive formation kinetics of the fcc and bcc phases. Near-equiatom Ni-V alloys were solidified via containerless processing methods and studied with electron microscopy techniques. TEM has shown the presence of a duplex structure of fcc and bcc in large (~100mg) droplet samples. TEM analysis has suggested that the duplex structure is not the result of a solid state transformation and that the fcc and bcc phases have apparently nucleated independently from the liquid phase. A thermodynamic model has been applied to the Ni-V system to calculate  $T_0$  temperatures for the relevant phases to map compositions and temperatures for which the duplex partitionless reaction is possible. A kinetic model has also been forwarded which takes into account both the nucleation and growth rates of the competing fcc and bcc phases. The nucleation kinetics analysis is in good agreement with experimental results. The model is currently undergoing further refinement to include transient nucleation effects as well as the influence of heat flow on microstructural development. Additional microstructural characterization will require cross-sectional TEM analysis of the containerlessly processed foil samples.

Near equiatom Mn-Al alloys represent an important class of permanent magnet materials. The key ferromagnetic phase is a metastable structure produced by solid state heat treatments. Recent drop tube and levitation melting studies have demonstrated for the first time that the metastable ferromagnetic  $\tau$  phase can be produced from the liquid provided high undercooling is achieved. With specially prepared samples it has been possible to access the thermodynamic driving forces involved in metastable  $\tau$  phase solidification. It also has been determined  $To^{\tau/d}$

temperature to help understand the metastable solidification reaction pathways which yield the metastable ferromagnetic  $\tau$  phase during the containerless processing. The attainment of the liquid undercooling required to nucleate the metastable phase from the melt is apparently facilitated by containerless processing. Additional experiments of large droplets will examine in detail the competitive solidification kinetics involved in sample size scale up in order to test and to extend the science base for a flight experiment. All expanded nucleation kinetics analysis of the small droplet samples will explore the phase selection between the  $\tau$  phase and an additional metastable product phase  $\gamma$ .

Building on the thermodynamic analysis a competitive nucleation model has been developed to account for the observed phase selection. As part of a test of the kinetics model a detailed statistical study of the metastable product yield as a function of sample size and processing gas, such as He and Ar gases, is underway. Furthermore, by optimizing the containerless processing conditions in levitation melting processing it has been possible to obtain essentially single phase samples of the  $\tau$  structure for scale up sample size (mm size) to approach bulk levels. In the above studies a new calorimetric system is being used to measure the temperature of falling drops during containerless processing to assess the complete thermal history.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	1	MS Degrees:	1
PhD Students:	1	PhD Degrees:	1

TASK INITIATION: 12/91    EXPIRATION: 11/94

PROJECT IDENTIFICATION: 963-25-07-06

RESPONSIBLE CENTER: JPL

### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Perepezko, J.H. Solidification reactions in undercooled alloys. Materials Science and Engineering, A179/180, 52 (1994).

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Kim, Y.J. and J. H. Perepezko. "Formation of a metastable ferromagnetic phase during containerless melt processing and rapid quenching in Mn-Al-C alloys." 31st Aerospace Sciences Meeting and Exhibit. American Institute of Aeronautics and Astronautics, (1993).

Perepezko, J.H. "Kinetic competition in undercooled liquids." International Symposium on Metastable Mechanically Alloyed and Nanocrystalline Materials, Grenoble France, June 1994.

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Perepezko, J.H. "Containerless processing for controlled solidification microstructures." MSAD Review, Huntsville, AL, May 1994.

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*Containerless Processing of Composite Materials*

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PRINCIPAL INVESTIGATOR: Prof. John H. Perepezko

University of Wisconsin, Madison

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The effects of containerless and low gravity processing are investigated in the synthesis of composite materials. Existing models predicting criteria for incorporation of particles dispersed in a melt are applied to metallic systems in a microgravity environment. The specific goals of the proposed research are to determine conditions necessary for an effective dispersion (i.e. uniform distribution of small particles), to characterize the bonding characteristics of particles with the matrix when processed under microgravity conditions; and to determine the implications of the results for effective solidification processing of composites. The ultimate goal of this work is to define conditions necessary for effective containerless or low-gravity processing as well as to determine characteristics which are unique to microgravity processing. Specifically, the results of this work will allow a critical test of existing theoretical models dealing with melt-particle interaction as well as processing conditions for particle incorporation. The outcome of these experiments will also further develop the basic understanding of relationships between microstructure and properties of metal matrix composites during microgravity processing.

**TASK DESCRIPTION:**

The focus of this study is on a model system of pure nickel with a discontinuous dispersion of reinforcement particles. The effects of gravity and containment on the microstructural development in particle reinforced composites is studied using free fall to offset the linear acceleration of gravity. Given the extensive knowledge base of the solidification behavior of nickel, assessment of melt and particle interaction becomes possible through a variety of means. The evaluation of the microstructure obtained during containerless processing allows for understanding of the conditions necessary a uniform distribution of particles without complications introduced by containing vessels such as chemical contamination or particle/mold interaction. When containerless processing is coupled with solidification during free fall, gravitational effects such as floatation, sedimentation, and density driven convection can be reduced. Variation of the magnetic field allows for vigorous mixing of the particles within the molten matrix, as well as solidification with or without external contact. The degree of undercooling may be controlled by monitoring the temperature with a two-color pyrometer and seeding crystallization with a needle when the desired temperature is reached. Through variation of the amount below the equilibrium freezing temperature a specimen is cooled, the driving force and therefore rate of solidification can be directly influenced. The varied solidification front velocity allows comparison to both predictions made by theoretical models as well as experimental data obtained without containerless or microgravity processing.

**TASK SIGNIFICANCE:**

Processing of particle reinforced composites under microgravity conditions can result in more uniform distributions of particles. With the reduction of the effects of gravity, problems inherent to conventional fabrication of metal matrix composites such as floatation, sedimentation, and density driven convection can be avoided. As has been demonstrated in directional solidification experiments, there is a critical rate at which solidification must occur for full incorporation (i.e. engulfment) of particles to occur. Particle engulfment into a forming solid in directional solidification experiments has been shown to depend on the temperature gradient, the solidification front velocity, and the size of the particles for a fixed chemical composition. The limitation in these experiments has been the container, which changes the shape of the front and which affects distribution of the particles through particle/surface interaction. Solidification under microgravity conditions permits a study of particle distribution without the effects of a container or gravity, which supplements information obtained from prior directional solidification experiments. As with all ground based microgravity experiments, this work serves as a guideline of steps to be followed in subsequent work actually conducted in space.

### PROGRESS DURING FY 1994:

The primary experimental facility, a ground based levitation system coupled with a laboratory scale drop tube, has been constructed and tested. Composite samples ranging from 1mm to 5mm diameter spheres are levitated, melted, and allowed to solidify during free fall. Alternately, the system also has the capability of melting and solidifying the specimen on a substrate, which allows the thermal history of the droplet to be recorded using a two-color pyrometer. Preliminary results indicate selection of nickel with aluminum oxide particles and nickel with titanium carbide particles are combinations at two extremes of surface energy balance. Aluminum oxide particles are typically excluded from the melt before solidification while the titanium carbide is almost exclusively incorporated. The cooling cycles which have been recorded are directly related to the resultant microstructures. Samples which cool below the equilibrium freezing temperature have a greater driving force for solidification and thus freeze at a higher rate than those which do not undercool. The changing rate of solidification is evident from the varied particle density across the composite sample upon metallographic examination of interior sections. The particle distribution as well as the cell morphology can indicate the comparative velocities during solidification of the droplet. These estimates of front velocity will be used to extend the experimental basis of support for existing models and possibly make additional constraints to their use.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 7/94 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-25-08-31

NASA CONTRACT NO.: NAG8-1068

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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Perepezko, J.L. "Containerless processing of particle reinforced composites." NASA Graduate Student Researchers Program Annual Symposium, Washington, DC, May 11-13, 1994.

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*Comparison of the Structure and Segregation in Dendritic Alloys Solidified in Terrestrial and Low Gravity Environments*

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PRINCIPAL INVESTIGATOR: Prof. David R. Poirier

University of Arizona

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CO-INVESTIGATORS:

J.C. Heinrich

University of Arizona

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**TASK OBJECTIVE:**

A primary goal of this research is to identify the growth conditions for microgravity experiments delineating the macrosegregation, microsegregation, and microstructure in comparison to the same features in directionally solidified alloys grown on earth with inevitable thermosolutal convection.

**TASK DESCRIPTION:**

Growth conditions are being defined by combining mathematical modelling and simulation with terrestrial experiments on binary metallic and model alloys. The investigation is carried forward at three universities, the University of Arizona with D.R. Poirier and J.C. Heinrich, Cleveland State University with S.N. Tewari, and Michigan Technological University with A. Hellawell.

**TASK SIGNIFICANCE:**

This research relates directly to almost all casting of metals as almost all practical cast metals are alloys which solidify under conditions of directional heat transfer. Alloys develop microstructures of dendrite primary and secondary arms, with micro- and macrosegregation patterns influenced not only by the basic physical processes of solidification but also by convective currents. To separate out the convective effects, microgravity experiments are necessary. Results of this work will eventually be included in numerical models for casting of alloys and will thereby impact alloys and will thereby impact a major portion of the economy.

**PROGRESS DURING FY 1994:**

Hypoeutectic Pb-Sn alloys, with compositions ranging from 10 to 58 wt. % Sn, have been directionally solidified at rates of 4 to 66  $\mu\text{m s}^{-1}$  in measured thermal gradients of 67 to 110  $\text{K cm}^{-1}$ . As shown in Fig. 1, this results in macrosegregation along the length of the directionally solidified alloys. In the absence of thermosolutal convection, the tin content along the length would have been uniform, except for an initial length of the order of mushy zone length.

Using the solutal build up between the fraction solids corresponding to 0.2 and 0.6 as a measure of the macrosegregation, it has been observed that the extent of macrosegregation increases with increasing tin content, becomes maximum for 33.3 wt. % Sn, and decreases with further increase in the tin content.

The intensity of the segregation along the length of the samples (and hence the presumed thermosolutal convection) has been explained in terms of a parameter which includes the primary arm spacing, the volume fraction interdendritic eutectic, and the eutectic and tip compositions in the liquid. Alloy composition, thermal gradient and solidification rate control primary arm spacing, fraction eutectic, and liquid concentration at the dendrite tips.

To date numerical simulations have been done using the thermal conditions and composition of one of the experimental ingots exhibiting "freckles" (also called "channel segregates"). As early as 1000 s after the onset of solidification, the numerical simulation shows upward flow next to the walls of the container, reminiscent of plumes that emanate upward from a remelted channel within a mushy zone. At 2000 s in the simulation, the channels along the walls are clearly evident and segregation is extensive, even though the leading part of the mushy

zone has advanced only 1 cm. For example, the concentration of solute in the remelted channels is as much as 31.5% Sn and, of course, some of the Sn is transported from the mushy zone into the all-liquid zone by the upward flowing plumes. With no convection, the simulation shows essentially no macrosegregation except for an initial portion where solidification was effected in a temperature field with a variable gradient.

We now have many experimental DS castings that have been prepared in measured or known temperature fields. The macrosegregation in each has been measured. In addition, microstructures have been taken from quenched ingots, including regions near the leading part of the advancing mushy zone. With these microstructures, we have been able to discern whether the solid solidified with a dendritic or cellular morphology. This is important because the extent of macrosegregation increases with decreasing growth speed as the solid changes from a dendritic to cellular morphology.

During the last six months of the grant, we plan to define the growth conditions for a microgravity experiment that could be done in an existing NASA furnace for a space flight experiment. To assist in defining growth conditions, we will run numerical simulations and study the effects of sample diameter, thermal gradient, solidification rate, and alloy composition. Hence, our next major emphasis will be on doing many more simulations based on the wealth of empirical data gained thus far for verification. After this is accomplished, we will simulate directional solidification of Pb-Sn alloys, under thermal scenarios that are achievable in a high gradient NASA furnace.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 2/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 963-25-05-09

NASA CONTRACT NO.: NAG3-1446

RESPONSIBLE CENTER: LeRC

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*Kinetics of Phase Transformation in Glass Forming Systems*

PRINCIPAL INVESTIGATOR: Dr. Chandra S. Ray

University of Missouri, Rolla

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objectives of this research are to: develop computer models for realistic simulations of nucleation and crystal growth in glasses, which would also have the flexibility to accommodate the different variables related to sample characteristics; and design and perform nucleation and crystallization experiments to verify these models. This research will lead not only to improved methods for the analysis of kinetic parameters for nucleation and growth determined from the peak profile studies of nonisothermal differential scanning calorimetry (DSC) or differential thermal analysis (DTA) measurements of crystallization, but also to determination of the relative merits and demerits of the theories presently used to study the phase transformations in glasses.

**TASK DESCRIPTION:**

This research is to study and explain the critical issues for the nucleation and crystallization in glass-forming systems. The reported data for the kinetic parameters that determine the overall nucleation and crystallization mechanisms are often difficult to interpret on the basis of existing theory. The interpretation becomes more difficult when a variation in the characteristics of the glass, such as the thermal history, composition, particle size of the sample, and concentration of the nucleating agent, are taken into account. This is probably due to the fact that the theories that are presently used to analyze the isothermal and nonisothermal crystallization data for glass-forming systems are over simplified. Glasses are traditionally prepared by cooling a melt and are not in a state of stable equilibrium. Consequently, phenomena such as atomic mobility, cluster distribution, nucleation and crystal growth rate, and viscosity pertaining to the nonequilibrium state need to be accounted for to establish an accurate description of the phase transformations in glass forming systems.

Glasses (primarily silicate based) that devitrify polymorphically by homogeneous nucleation will be studied experimentally and by computer modeling. A lithium-disilicate ( $\text{Li}_2\text{O} \cdot 2\text{SiO}_2$ ) glass will be used for most calculations and experimental measurements since the necessary thermodynamic and kinetic parameters are available for this system. Other glasses that will also be investigated to verify the general applicability of the model include soda-lime-silica ( $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 3\text{SiO}_2$ ) and barium-disilicate ( $\text{BaO} \cdot 2\text{SiO}_2$ ). Crystallization experiments for the glasses will be conducted by DTA or DSC using the conditions used for computer modeling, such as the quench rate used to prepare the glass, sample weight, particle size of the sample, precrystallization heat treatment temperature and time, type and amount of nucleating agent, and DTA or DSC scanning rate. The experimental results will be compared with those predicted by the model. If flight opportunities become available, glasses of identical compositions will be prepared in space, and the same crystallization experiments as were conducted for the Earth-melted control samples will also be performed for the returned flight samples. The results from this research are anticipated to yield a realistic model for nucleation and crystal growth processes occurring in glass-forming melts which would provide not only an improved scientific understanding for these processes, but also allow a more accurate quantitative analysis of the thermal analysis data. This would help to explain several anomalous experimental results obtained for the kinetic parameters for crystallization and would lead to values that are more physically interpretable. The relative role of heterogeneous and homogeneous nucleation on glass formation can be determined, and the reported observation that a glass prepared in low gravity is more homogeneous and more resistant to crystallization than an identical glass prepared at 1-g can be explained.

**TASK SIGNIFICANCE:**

The results from this research are anticipated to yield a realistic model for nucleation and crystal growth processes occurring in glass-forming melts, which would provide not only an improved scientific understanding for these processes, but also allow a more accurate quantitative analysis of the thermal analysis data. This would help to explain several anomalous experimental results obtained for the kinetic parameters for crystallization and would lead to values that are more physically interpretable. The relative role of heterogeneous and homogeneous nucleation on glass formation can be determined, and the reported observation that a glass prepared in low gravity is more homogeneous and more resistant to crystallization than an identical glass prepared at 1-g can be explained.

**PROGRESS DURING FY 1994:**

1. An experimental method that uses differential thermal analysis (DTA) was developed for determining the nucleation rates at different temperatures for a lithium disilicate ( $\text{LS}_2$ ) glass. The DTA peak height for homogeneously nucleated  $\text{LS}_2$  glasses was compared with that for glasses containing different amounts of platinum. The only assumption made in developing this method was that the crystal growth rate on a heterogeneous nucleus was essentially same as that on a homogeneous nucleus. Nucleation rates for the  $\text{LS}_2$  glass determined by the present method are in excellent agreement with those reported in the literature. The validity of this method is now being tested using computer modeling developed by Dr. K. F. Kelton at the Washington University in St. Louis.
2. The effect of Particle size on DTA peak parameters (height, temperature and width) was experimentally established for the  $\text{LS}_2$  glass. Analyzing the data as a function of particle size, it is possible to predict the dominant growth mechanism (bulk or surface) that occurs during crystallization of a glass. The DTA peak profiles for the  $\text{LS}_2$  glass as a function of particle size indicated a considerable contribution of surface crystallization, which was also observed in the scanning electron microscopy (SEM) for the crystallized glass particles. The computer programs developed by Dr. K. F. Kelton were modified to take account the effect of surface crystallization, and a significant improvement in the calculated DSC peak profiles was observed for the  $\text{LS}_2$  glass.
3. An experimental method was developed to determine the nucleation rate-like curve for the  $\text{LS}_2$  glass from dielectric constant measurements. The dielectric constant was measured at 1000 kHz for samples of about 2 mm thick and 8 mm in diameter after prenucleation and crystallization treatment at different temperatures for different times. The plot for the inverse of dielectric constant as a function of nucleation temperature yields a curve similar to that of the nucleation rate curve for the  $\text{LS}_2$  glass.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 12/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 962-26-08-08

NASA CONTRACT NO.: NAG8-898

RESPONSIBLE CENTER: MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1994:****Presentations**

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Ray, C.S. "Kinetics of phase transformations in glass forming melts." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.



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*The Effects of Microgravity on Vapor Phase Sintering*

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PRINCIPAL INVESTIGATOR: Prof. Dennis W. Readey

Colorado School of Mines

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The objective of this research is to develop an improved understanding of solid sintering by investigating the effects of reactive atmospheres and vapor transport on the sintering of ceramics. Ceramic systems that have been studied to date show a wide range of behavior that is thought to be due to the close proximity of particles in powder compacts appropriate for sintering and densification. The microgravity environment offers the unique opportunity to compare behavior of dispersed particles, or particles with minimal agglomeration, with those in a dense powder compact. These comparisons allow the separation of the relative contributions of particle interaction by gas phase diffusion and coalescence controlled by grain boundary mobility.

**TASK DESCRIPTION:**

This research combines a study of the effects of vapor transport in powder compacts with those of dispersed particles. Ideal systems for space-based experiments will be determined from initial ground-based research. First, such an ideal system must exhibit significant microstructural effects of vapor transport. Second, the ideal system must show a difference in behavior between constrained particles in close proximity such as those in a typical ceramic powder compact compared to dispersed, unconstrained particles. Finally, an ideal system would be one that could be used at low temperatures, would not involve toxic materials or atmospheres, and would be amenable to short-time experiments. The latter would allow experiments to be performed in drop tower or aircraft parabolic maneuvers.

**TASK SIGNIFICANCE:**

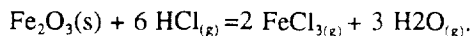
The significance of this research is that it will lead to:

1. An improved understanding of solid state sintering of ceramics.
2. An improved understanding of the contributions that various transport processes make to microstructure development during the sintering of ceramics.
3. Will provide information for the design and production of porous ceramics through enhanced vapor phase sintering.

The latter is particularly important from a technical point of view since materials such as  $\text{Al}_2\text{O}_3$  are being sintered to a controlled pore size and porosity for filter applications and for infiltration to form interpenetrating ceramic-metal composites.

**PROGRESS DURING FY 1994:**

In virtually all the systems studied, the most significant result is that the main effect of enhanced vapor transport on sintering is significant particle coarsening with virtually no densification. The results of  $\text{Fe}_2\text{O}_3$  in HCl illustrate the point. In HCl, the following reaction dominates the transport species in the gas phase:



This results in grain growth with essentially no shrinkage. The particles grow with very close to a  $t^{1/3}$  dependence and the particle size is within a factor of two calculated from classical Ostwald ripening models.<sup>1, 2, 3</sup>  $\text{Fe}_2\text{O}_3$  fits the model of coarsening by vapor phase diffusion reasonably well. However,  $\text{Al}_2\text{O}_3$ , which reacts in a similar fashion in HCl, does not follow a  $t^{1/3}$  dependence, the particle size is much smaller than predicted by the models, and the apparent activation energy for the process is almost an order of magnitude higher than what would be expected. The reason that  $\text{Al}_2\text{O}_3$  is thought to not fit the model is that coarsening is inhibited in this system because of low grain boundary mobility. Therefore, even with rapid vapor transport, coarsening cannot occur unless grain boundaries are mobile. This is due the constraints on a particle in powder compact by its nearest neighbor particles. If the particles were not constrained but dispersed, boundary mobility would not be a necessary condition for coarsening.

#### MICROGRAVITY EXPERIMENTS

The potential for microgravity in these studies is to provide an environment where particle coarsening can take place under relatively dispersed conditions and compare the results of coarsening of dispersed particles with those in a powder compact. Of course, some agglomeration of these small powder particles will occur in microgravity. However, the degree of agglomeration and the constraints of nearest neighbors will be minimized compared to the constraints of the typical compacted powder during sintering.

The times for previous coarsening experiments have been as long as hundreds of hours. This implies that for these experiments to be successful, shuttle flights will be necessary. As a result, it is desirable to eliminate toxic or explosive atmospheres such as HCl and hydrogen. Furthermore, experiments at lower temperatures are much more amenable to microgravity experiments than the 1200°C to 1600°C temperature range that has been used in the past for experiments with ceramic powders. As a result, it may be possible to use a low temperature surrogate to investigate the same phenomena that are important at high temperatures in toxic atmospheres in ceramics. The desirable characteristics of such a low temperature surrogate are:

- high vapor pressure
- low melting point
- simple crystal structure
- non-toxic
- stable in air
- small particle size
- shows particle growth and no densification

An additional benefit of low temperature surrogates would be the ease of measuring the gas diffusion coefficient and the surface energy. These are two important parameters in vapor transport that are difficult to obtain for high temperature ceramic materials and are normally estimated. Therefore, the initial phase of this research has focused on finding suitable low temperature surrogate materials, both inorganic and organic.

#### INORGANIC SURROGATES

A list of some 50 potential inorganic compounds was compiled from the literature based on their melting points and vapor pressures. The following systems were chosen for further experimental investigation based primarily on their vapor pressures, melting points, and relative lack of toxicity. Iron Chloride,  $\text{FeCl}_3$  has a melting point of 304°C and a vapor pressure above 100 Pa above 200°C. However, even in the arid Colorado climate, dry powder quickly absorbed sufficient atmospheric moisture to become a puddle in only a few minutes. It was eliminated from further study. Aluminum Chloride,  $\text{AlCl}_3$  is similar to  $\text{FeCl}_3$  subliming at 180°C and the exposed powder did not seem to be adversely affected by atmospheric moisture. However, x-ray diffraction showed that the chloride was reacting with the atmosphere to produce what were probably hydrated chlorides or hydroxides. It too was eliminated from further study. Iodine,  $\text{I}_2$  has a melting point of 112.9°C and a vapor pressure above 100 Pa above 38°C. However, this vapor pressure is so high that it precludes observation in an ordinary SEM which is necessary to evaluate particle growth in a powder compact. In addition, powders were not easily made so iodine was eliminated from

further study. Ammonium bromide and ammonium chloride,  $\text{NH}_4\text{Br}$ , and  $\text{NH}_4\text{Cl}$  are very similar in behavior and offer the additional advantage of being able to form solid solutions to a greater flexibility in properties.  $\text{NH}_4\text{Br}$  sublimates at  $396^\circ\text{C}$  and has a vapor pressure in excess of 100 Pa above  $195^\circ\text{C}$ .  $\text{NH}_4\text{Cl}$  sublimates at  $340^\circ\text{C}$  and has a vapor pressure in excess of 100 Pa at  $160^\circ\text{C}$ . The as-received powders had a particle size greater than 100 micrometers and ball milling was not very successful in reducing the particle size due to the plasticity of these compounds. Small particle size powders could be made by evaporation and condensation and by reacting  $\text{NH}_3$  and  $\text{HCl}$ . Loose powders were heated for various times at various temperatures and some particle growth was observed. Sodium chloride,  $\text{NaCl}$ , is typical of most of the alkali halides in that its vapor pressure at its melting point of  $801^\circ\text{C}$  is only about 10 Pa, very near the lower limit where significant vapor transport might be expected. However,  $\text{NaCl}$  was chosen for further study since previous sintering studies have suggested that vapor transport was an important transport process during sintering. Preliminary experiments on loose powders do indicate particle growth but powder compacts shrink considerably. This indicates that vapor transport does not dominate solid diffusion processes and probably eliminates  $\text{NaCl}$  from further consideration.

### ORGANIC SURROGATES

Over thirty organic compounds were found in the literature that had high vapor pressures at low temperatures. Many were eliminated because of their alleged toxicity and others simply because their melting points were too low or their vapor pressures too high. An example of one such material is  $\text{H}_2\text{O}$ , ice. Although it has many desirable features for this study, handling it below room temperature creates problems that are to be avoided.

Two systems were chosen for further study. Salicylic acid,  $\text{C}_7\text{H}_6\text{O}_3$ , has a melting point of  $159^\circ\text{C}$  and a vapor pressure of 100 Pa at  $113^\circ\text{C}$ . Trans-cinnamic acid,  $\text{C}_9\text{H}_8\text{O}_2$ , which has a melting point of  $133^\circ\text{C}$  and a vapor pressure of about 100 Pa at  $127^\circ\text{C}$ . Both of these materials could be successfully ball milled to produce small particle size material for initial screening experiments. However, the very high vapor pressures of these materials has precluded their investigation by SEM. This has made particle size analysis considerably more difficult. A satisfactory replica technique has now been found and microstructure changes in powder compacts are now being studied. Particle growth of loose powders has been observed and has been analyzed by optical microscopy.

### CONCLUSIONS

The necessity for long flights and non-toxic atmospheres has led to a search for suitable low temperature surrogates to study vapor phase sintering of ceramics. There are several inorganic and organic compounds that appear promising. The very high vapor pressures at low temperatures for some of these compounds preclude the use of standard electron microscopy for analyzing the particle growth, either dispersed or in powder compacts.

### FUTURE WORK

Particle growth experiments will continue with ammonium chloride, salicylic acid, and cinnamic acid in both compacts and with powders dispersed on glass wool. In addition, experiments are being initiated with  $\text{MgO}$ ,  $\text{ZnO}$ , and  $\text{Al}_2\text{O}_3$  powders in compacts and dispersed on graphite in both  $\text{HCl}$  and inert atmospheres. These systems are known to undergo particle growth in  $\text{HCl}$  in a powder compact. To be appropriate for microgravity experiments, they must show greater growth as dispersed particles on the graphite compared to the constrained particles in a powder compact.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 2  
PhD Students: 0

TASK INITIATION: 12/92 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 962-26-05-08

NASA CONTRACT NO.: NAG3-1409

RESPONSIBLE CENTER: LeRC

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*Modeling of Detached Solidification*

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PRINCIPAL INVESTIGATOR: Dr. Liya L. Regel

Clarkson University

CO-INVESTIGATORS:

W.R. Wilcox

Clarkson University

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**TASK OBJECTIVE:**

The long term objective of the proposed research is to develop techniques to realize detached solidification reliably, in order to produce crystals with fewer defects. To achieve this objective, it is necessary to understand the mechanism for detached solidification. It is the primary objective of the research to further this understanding.

**TASK DESCRIPTION:**Theory

A quantitative theory for detached solidification will be developed. Because the radius of curvature of the ampoule is large compared to that of the meniscus, we will assume the ampoule wall is flat and of infinite extent. That is, we reduce this to a two dimensional problem. The pressure difference across the meniscus is related to its curvature by the Laplace equation. In fact the radius of curvature must be constant, so that the meniscus is an arc of a circle. The meniscus contacts the ampoule wall at the equilibrium contact angle. The meniscus angle at the corner of the growth interface is arbitrary, but determines the growth direction. Thus one can assume an infinitesimal gap as a beginning and easily trace the path of the growth interface for given values of melt-ampoule contact angle, melt-vapor surface tension, pressure difference, and the angle between the growth direction and the meniscus at the freezing interface. We will develop an equation for the steady state gap width. From an estimate of the minimum stable gap width, we will be able to specify the conditions under which detached growth occurs.

One critical parameter is the pressure difference across the meniscus. We will estimate this pressure difference based on a numerical calculation of diffusion of dissolved gas as it is rejected by the growing solid. We will consider a finite ampoule diameter, an ideal mixture, and an ideal gas. This will enable us to estimate the residual acceleration (g level) required in order to achieve detached growth.

Experiments

As a consequence of our new understanding of detached growth in space, we have developed a concept for achieving it on earth. Basically, a melt is supported by a piston at the bottom, in order to provide the pressure difference condition required to give an appropriate meniscus shape and gap width. Because of the density change and the diameter change that occur upon freezing, the piston must be moved continuously to maintain the proper meniscus shape.

This has some resemblance to Czochralski growth, except that the solid is sealed in the ampoule and it may be more difficult to control the crystal diameter. What we propose doing is a simple proof of concept experiment with a low melting metal, such as tin. This will enable us to observe optically the solid detachment, meniscus formation, and solidification. A simple upside-down Bridgman apparatus will be fabricated with transparent heaters. The piston will be driven by a motor whose rate of motion is controlled by the human observer. The heaters will be lowered by another motor in order to cause solidification downward. The interfacial region will be videotaped through an appropriate lens.

Temperature measurements will be made in the ampoule during some runs. Thermocouples will be inserted from above and held in fixed positions during the run.

A flight experiment will be planned to test our model. If our model is correct, silicon will not display detached growth in space. Although the contact angle of molten silicon on quartz is large, the adhesion of solid silicon to quartz is extremely strong.

### TASK SIGNIFICANCE:

Solidification in space has often yielded crystals that were smaller in diameter than their containing ampoules. When this occurs, crystals of much higher perfection are produced. We now understand how this occurs and why crystal perfection is greater. This research project aims to put this understanding on a firm basis so that detached solidification can always be achieved. The research should also yield improvements in crystal growth on earth.

### PROGRESS DURING FY 1994:

1. Recruited M.S. student in chemical engineering in September 1994.
2. Began derivation of equations for meniscus shape.
3. Began design of experiment.

### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 0

TASK INITIATION: 6/94 EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-21-08-21

NASA CONTRACT NO.: NAG8-1063

RESPONSIBLE CENTER: MSFC

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*Electrostatic Containerless Processing*

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PRINCIPAL INVESTIGATOR: Dr. Won-Kyu Rhim

Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The Electrostatic Containerless Processing Task objective is to investigate thermophysical properties, nucleation kinetics, and metastable phase selection from undercooled metals, semiconductors, and glasses by utilizing the advanced capabilities of isolating molten drops in a clean environment provided by the High Temperature High Vacuum Electrostatic Levitator.

**TASK DESCRIPTION:**

The High Temperature High Vacuum Electrostatic Levitator (HTHVESL) has been developed at JPL and has important capabilities for the containerless materials science research. There are other methods of isolating liquid drops such as the electromagnetic and acoustic levitation methods. However, the HTHVESL combines the following important capabilities required for the various thermophysical property measurements and for the studies of solidification processes: (a) it provides quiescent and clean processing environment without inducing convective flow in the melt, (b) both conducting and non-conducting materials can be levitated, and (c) sample temperature can be independently controlled without being affected by the levitation force. With additional capabilities of measuring temperature, specific heat to hemispherical total emissivity ratio, thermal expansion coefficient, surface tension, viscosity, and electric conductivity, this system is well suited for the containerless materials science research both in the ground laboratories as well as in the micro-gravity environment.

**TASK SIGNIFICANCE:**

The capability of levitating molten metals, semiconductors, and insulators in a clean and quiescent environment allows us to investigate various thermophysical properties such as specific heat, total hemispherical emissivity, thermal expansion coefficient, surface tension, viscosity, electric and thermal conductivity of undercooled melts. In particular, the capability of processing semiconductors and insulators is very unique to the present approach. Investigation of nucleation and solidification processes will not only increase knowledge which is fundamental in physical processes it will also have important engineering application in designing a certain microstructure or a metastable state (including amorphous phase).

**PROGRESS DURING FY 1994:****Major Technical Achievements:**

A significant breakthrough has been made in the high temperature electrostatic levitation technology. About 3 mm size samples with varying density could be levitated against gravity with excellent stability and the sample could be subjected to a required thermal cycle in a high vacuum environment. For example, high temperature processing has been demonstrated by allowing a number of nickel ( $T_m=1728$  K) and zirconium ( $T_m=2128$  K) samples to go through melting-superheating-undercooling-solidification cycles. Processing even higher  $T_m$  materials will be achieved if the present heating system is improved.

The High-Temperature High-Vacuum Electrostatic Levitation Facility (HTESL) at JPL has been developed under NASA's Advanced Development Activity (ADA) program, and it is being recognized as one-of-a-kind in the world which is able to meet various challenges imposed by the high temperature containerless materials processing community both at universities as well as industrial laboratories.

The high temperature electrostatic levitator provides the capability of superheating, undercooling, and solidifying pure metals, alloys, semiconductors, glasses, and ceramics in a clean and quiescent environment. When a levitated sample is melted, one can repeat various pre-programmed thermal cycles using a single sample, observing different response in each thermal cycle. The advantages of the HTESL over conventional electromagnetic levitators (EML) are that: (i) it can accommodate a broad range of materials, including metals, semiconductors and insulators, (ii) sample heating and levitation do not interfere with each other so that the sample temperature can be varied over a wide range (from room temperature up to 2000°C using a presently available heating source), (iii) employment of feedback control provides quiescent sample positioning during processing which allows to measure accurate temperature and sample volume, (iv) sample processing is done in a high vacuum ensuring purity of samples, and (v) it provides a widely open sample view for non contact diagnostic measurements.

Various sample materials have been successfully melted during levitation, for example, pure elements such as Zr, Ni, Si, Cu, Ge, Al, Pb, Bi, Sn, In, and various alloys and nonconducting glasses/ceramics have been melted, and thermophysical properties of several of these materials have been determined. Specific technical capabilities we have developed are listed below.

Specific heat/hemispherical total emissivity: Specific heat is a very important thermodynamic parameter from which all other parameters such as enthalpy, entropy, and Gibbs free energy can be determined. These quantities measured in undercooled regions of various materials carry special implications for the studies of solidification processes and for the selection of useful metastable phases. Purely radiative cooling provided by the HTESL allows us accurate measurements of the ratio between specific-heat and hemispherical-total-emissivity. Therefore, availability of one of these parameters will determine the other. Such measurements have been demonstrated in nickel, zirconium, silicon and germanium over a wide temperature range around their melting points.

Undercooling and nucleation: Since samples are processed in a high vacuum, greater undercooling can be achieved. Furthermore, since there is no appreciable internal flow induced by the levitation force, one can safely rule out possible dynamic nucleation. Indeed, our undercooling/nucleation studies consistently revealed single nucleation mechanism in contrast to other equivalent data obtained either by an electromagnetic levitator or a drop tube. Combination of a high speed imaging system and a pyrometer will allow us to measure the solidification velocity upon nucleation.

Metastable phases: Containerless approach of solidification using HTESL will be an effective way of achieving high undercooling in molten samples, thereby obtaining metastable phases. Specific metastable phase may be selected by injecting a seed particle having a right phase. We have demonstrated metallic glass formation in some of glass-forming alloys measuring critical cooling rate and determining the Temperature-Time-Transformation (T-T-T) curves. Developing new glass forming alloys using HTESL will be a very productive area for the development of industrially useful new materials.

Surface tension: Spherical drop shape of melts levitated by HTESL greatly simplify surface tension measurements with increased accuracy. In view of the fact that surface tension is particularly sensitive to even minute surface contamination, HTESL operating in a high vacuum environment would be an ideal approach to the surface tension measurements particularly of those chemically reactive high temperature materials. We have demonstrated surface tension measurements in various pure metals, alloys, and semiconductors with great accuracy.

Thermal expansion coefficient measurement: Accurate determination of mass density increases the accuracy of other thermophysical parameters such as specific heat, surface tension, thermal diffusivity, kinematic viscosity etc. Quiescent levitation as well as nearly spherical (or at least axi-symmetric) sample shape provided by HTESL offer an ideal situation for the measurements of mass densities as well as thermal expansion coefficients. An optical system which produces high-magnification/ high-contrast video images was set up, and image processing software was developed which measures the sample volume within 1% error. We are in the middle of testing this capability using high temperature liquid samples.

Semiconductor processing: We have succeeded in processing both pure silicon and germanium samples. Having isolated from container walls the silicon melt showed undercooling as deep as 250 K. Capability of processing semiconductor materials for thermophysical property measurements is particularly important since these parameters are needed in numerical modeling of crystal growth environment. We have also succeeded in measuring surface tension of silicon melts over 400 K around the melting temperature.



Viscosity: HTESL promises an important possibility of measuring viscosity by observing the free damping time of an oscillating drop. This will be possible only in the absence of appreciable internal flow as much as any interference by the levitation forces. A preliminary attempt for viscosity measurement using a tin sample has revealed that greater caution is required in order to increase measurement accuracy. Development of the viscosity measurement technique using HTESL is in progress.

Electrical and thermal conductivity: Some feasibility study have been conducted with promising conclusion, however, development of these techniques will be subject to future funding.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/89 EXPIRATION: 9/94

PROJECT IDENTIFICATION: 962-25-04-08

RESPONSIBLE CENTER: JPL

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Rulison, A. and W. K. Rhim. Specific heat to hemispherical total emissivity ratio for undercooled liquid nickel, zirconium, silicon, and germanium. Metall. Trans. B. (1994 in press).

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Kim, Y.J., R. Busch, W. L. Johnson, A. J. Rulison, and W. K. Rhim. "Undercooling studies of the bulk metallic glass forming  $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10.0}Be_{22.5}$  alloy during containerless electrostatic levitation processing." 95th TMS Annual Meeting, Feb. 12-16, 1994, Las Vegas, NV.

Rhim, W.K. "High temperature ESL." 6th Space Station Utilization Workshop, Jan. 18-19, 1994, Tokyo, Japan.

Rhim, W.K. "Materials processing by high temperature electrostatic levitation." Canadian Round Table Workshop on Microgravity Containerless Processing, Feb. 24 - 25, 1994, St. John's, Newfoundland, Canada.

Rhim, W.K. and A. J. Rulison. "Thermophysical property measurements of undercooled silicon." 12th Symposium on Thermophysical Properties, June 19-24, 1994, Univ. Colorado, Boulder, Colorado.

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Szekely, J., W. K. Rhim, J. Watkins. "Critical assessment of electromagnetic and electrostatic levitation for containerless processing in microgravity and under earthbound conditions." 123rd TMS Annual Meeting, Feb. 27-Mar. 3, 1994, San Francisco, California.

#### Patent

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*Drop Tube Operation*

PRINCIPAL INVESTIGATOR: Dr. Michael B. Robinson

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective is to maintain and upgrade the operation capability of and to conduct experiments in the MSFC 105-meter drop tube. The drop tube facility includes, in addition to the tube itself with the associated pumps and valves, such items as furnaces, levitators, other sample holding or handling devices, and data recording systems. This research includes the operation of a facility laboratory located at the facility. This laboratory includes furnace test facilities as well as pyrometer calibration platforms.

**TASK DESCRIPTION:**

This research covers work in the area of defining, developing, and conducting experiments using the low-gravity capabilities of the drop tube. Such experiments may be in themselves complete investigations to develop new knowledge or to prove theories, or they may serve as precursors for more extensive experiments to be conducted in space. This research also includes studies and experiments to define the effects of various levels and durations of acceleration perturbations on microgravity experiments.

The research approach will be to:

- Study the limits of undercooling in a low-gravity containerless environment and ascertain if nucleation occurs homogeneously at the undercooling limits.
- Evaluate the effects of deep undercooling by containerless processing on resulting microstructure and define and understand the types of phases formed, their shapes and sizes, and their distribution, abundance, composition, homogeneity, and substructure.
- Study the formation of quasicrystalline material through deep undercooling.
- Study the spreading of liquids as a function of undercooling in order to better understand the thermophysical properties of materials.

**TASK SIGNIFICANCE:**

This research activity is an essential part of a successful program of research in microgravity science and applications. Many experiments proposed for flight on the Space Transportation System (STS) can be developed and tested in preliminary form using drop facilities. This can result in significant savings through the proving of experiment concepts and equipment designs before proceeding to much more costly space flight hardware. It also provides additional data that can be compared with data obtained from the space flight experiments. And in some cases, the data obtained from the drop tube or aircraft experiments prove to be sufficient to satisfy the experimenter's requirements, thus obviating the need to proceed with an experiment on the STS. The result is an overall savings in the cost of conducting microgravity experiments while adding to the scope and quality of the results obtained.

**PROGRESS DURING FY 1994:**

During FY94 approximately 400 samples were processed, the majority of which was in support of the IML-2 flight of TEMPUS. In addition, numerous improvements were implemented, including the installation of a black tube liner which will eliminate stray reflections. These reflections are a major source of error in nucleation temperature measurements.

**STUDENTS FUNDED UNDER RESEARCH:**

TASK INITIATION: 1/78 EXPIRATION: 1/95

PROJECT IDENTIFICATION: 963-80-10-02

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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**Presentations**

Bayuzick, R.J., Hofmeister, W.H., Morton, C.W., and Robinson, M.B. "Effects of nucleation by containerless processing." NASA Microgravity Materials Science Conference, Huntsville, AL, May 24-25, 1994.

Rathz, T.J., Weimer, J.J., and Robinson, M.B. "Undercooling of immiscible metal alloys without crucible-induced wetting and nucleation." Alabama Materials Research Conference, Tuscaloosa, AL, September, 1994.

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

### *Measurement of the Optical and Radiative Properties of High-Temperature Liquid Materials by FTIR Spectroscopy*

PRINCIPAL INVESTIGATOR: Dr. Michael B. Robinson

NASA Marshall Space Flight Center (MSFC)

#### CO-INVESTIGATORS:

S. Khrishan

CRI

T. Rathz

University of Alabama, Huntsville (UAH)

G. Workman

University of Alabama, Huntsville (UAH)

#### TASK OBJECTIVE:

The objective is to fully develop the use of FTIR spectroscopy for the purpose of determining, at a high rate of speed, the normal and total hemispherical emissivities of deeply undercooled materials over the wavelength range of 2 to 20 microns. Due to the nature of the approach, the spectral emissivities can be determined quickly over a wide wavelength range and applied over a wide temperature range.

#### TASK DESCRIPTION:

The task will involve development of the technique of measuring the radiative properties of high-temperature, highly reactive materials in the liquid and undercooled liquid state by use of FTIR spectroscopy. The sample will be positioned in a Containerless environment by either electromagnetic or electrostatic positioners. The technique offers the advantage of fast measurement so that sample temperature stability will not present insurmountable problems.

#### TASK SIGNIFICANCE:

Optical property measurements are essential to the understanding of the behavior of liquids at high temperature. Accurate knowledge of these properties provide an ability to validate theories of nucleation, solidification, and undercooling; and provide the basis for accurate non-contact temperature measurement. They are particularly needed for accurate measurement of high temperatures so that existing and new thermophysical property measurements can be interpreted correctly.

#### PROGRESS DURING FY 1994:

All necessary equipment has either been designated or ordered. Designs have been finalized and construction of the FTIR system and processing chamber should begin shortly.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-25-08-32

NASA CONTRACT No.: In-house

RESPONSIBLE CENTER: MSFC

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*Undercooling Behavior of Immiscible Metal Alloys in the Absence of Crucible Induced Nucleation*

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PRINCIPAL INVESTIGATOR: Dr. Michael B. Robinson

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

D. Frazier

NASA Marshall Space Flight Center (MSFC)

B. Facemire

NASA Marshall Space Flight Center (MSFC)

T. Rathz

University of Alabama, Huntsville (UAH)

G. Workman

University of Alabama, Huntsville (UAH)

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TASK OBJECTIVE:

The objective of this effort is to determine if processing immiscible metals in a containerless environment would alter the critical point wetting mechanism and the extent to which this would lead to changes in the subsequent nucleation kinetics. If the nucleation kinetics are suppressed enough to allow bulk undercooling, the nucleation recalescence will be measured. In addition, the droplet surface conditions can be varied by processing in a vacuum versus a gas environment to alter the surface composition and thereby the wetting potential of the fluid phases.

## TASK DESCRIPTION:

Research in a containerless, low-gravity environment provides much information as to whether preferential wetting of the free surface occurs and more importantly whether it can be controlled. The MSFC 105 Meter Drop Tube Facility will provide the low-gravity, containerless environment necessary for this study.

## TASK SIGNIFICANCE:

Cooling of monotectic alloys into the miscibility gap will lead to nucleation of droplets with in the liquid matrix. Previous earth and space experiments have concentrated on the morphology of the bulk microstructure by attempting to control the convective-diffusive flow of these droplets with the proper selection of the crucible material and thermal fields. Immiscible metal systems have as yet not been studied in a containerless environment, eliminating crucible induced flows,

## PROGRESS DURING FY 1994:

Six different systems have been identified for initial investigation. Starting samples have been generated and will be soon processed in the Drop Tube Facility. Preliminary investigations of the microstructures of the as cast materials has begun.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-25-08-33

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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*Undercooling Limits in Molten Semiconductors and Metals: Structure and Superheating Dependencies*

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PRINCIPAL INVESTIGATOR: Dr. Frank G. Shi

University of California, Irvine

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## CO-INVESTIGATORS:

W.K. Rhim

Jet Propulsion Laboratory (JPL)

A.J. Rulison

John Hopkins University

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**TASK OBJECTIVE:**

The objective of this work is to study the onset of nucleation of crystals and thus the undercooling limits for melts of semiconductors and metals in order to experimentally test and further develop a model for the onset of nucleation.

**TASK DESCRIPTION:**

The undercooling experiments are performed using the existing High Temperature Electrostatic Levitator. The HTESL is fully operational and has proven to be effective in melting and undercooling metals, e. g. zirconium and nickel, semiconductors, e. g. germanium, and ceramics, e. g. silicon dioxide based glass-ceramics. The HTESL is fitted with a single color pyrometer which are used to measure the undercooling limits of all the samples.

**TASK SIGNIFICANCE:**

The onset of nucleation in the undercooled liquids and thus the achievable undercooling level determines the selection of the final microstructures and formation of metastable amorphous phases. The ability to predict and control the onset of nucleation is therefore important in advanced materials processing.

**PROGRESS DURING FY 1994:**

The necessary contractual arrangements are still being processed to fund this project which was accepted under the 1993 NRA-93-OSSA-12.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-21-08-22

NASA CONTRACT No.: NAG8-1082

RESPONSIBLE CENTER: MSFC

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*Crystal Nucleation, Hydrostatic Tension, & Diffusion in Metal and Semiconductor Melts*

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PRINCIPAL INVESTIGATOR: Prof. Frans A. Spaepen

Harvard University

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## CO-INVESTIGATORS:

M.J. Aziz

Harvard University

D. Turnbull

Harvard University

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**TASK OBJECTIVE:**

The objective is to develop basic understanding of the phenomena and processes that are central to the microgravity program: crystal nucleating, glass formation, and diffusion in the liquid state.

**TASK DESCRIPTION:**

Crystal nucleation is studied in elemental metal, semiconductor, or quasi-crystal-forming droplets coated with different fluxes, droplets with clean surfaces in vacuum, and droplets solidified in a drop tube. The effect of hydrostatic stress on the nucleation kinetics is studied by dilatometry. The crystal-melt interfacial tension is studied experimentally and theoretically. The diffusivity in the liquid state is measured from the broadening of impurity profiles after pulsed laser melting.

**TASK SIGNIFICANCE:**

Studies of the undercooling of liquids and the kinetics of crystal nucleation are an important category of experiments that exploit the containerless environment provided by microgravity. Our work, ground-based, is aimed at advancing the understanding of the fundamentals of undercooling, nucleation, and glass formation; at exploring the potential and limitations of ground based alternatives such as fluxing and drop-tube processing; and at exploring the potential of ground-based containerless processing facilities provided by the microgravity program.

**PROGRESS DURING FY 1994:**

Work on the undercooling of semiconductor melts is continuing. Undercooling experiments on silicon droplets have been performed in a variety of oxide and halogen fluxes. Much effort was devoted to the development of an appropriate flux. Chloride and fluoride fluxes were too volatile at the high temperatures required to melt silicon. Many oxide fluxes, such as  $B_2O_3$  which is very effective for undercooling liquid germanium, are reduced by silicon, and as a result lose their effectiveness. This leaves  $SiO_2$ ,  $CaO$ ,  $BaO$ ,  $Al_2O_3$ ,  $TiO_2$ , and  $MgO$  and few others. At the same time, the flux must be sufficiently fluid at the undercooled temperatures to be effective.

A systematic study of the ternary and quaternary alloys of the chemically compatible oxides revealed that molten 47.5%  $SiO_2$ , 39%  $BaO$ , 13.5%  $CaO$  was an appropriate flux. The first three undercooling experiments gave two large undercoolings of 350 K and 310 K. These are the largest undercoolings measured in bulk liquid silicon; the previous maximum undercooling was 285 K. This measurement requires a re-interpretation of the agreement claimed between the observations of bulk undercooling and of undercooling following pulsed laser melting.

In a parallel series of experiments, melting and resolidification of uncoated silicon and germanium on different substrates were investigated with the aim of developing a systematic understanding of the effects of heterogeneous nucleants. Specific substrates included (with undercoolings listed): amorphous silica (190-230 K), crystalline silica (quartz) (60-160K), sapphire (30-55K). Substrates used for germanium included amorphous silica (120-170K), crystalline silica (quartz) (110-140K), sapphire (70-120K), mica (140-150K), silicon (9-15K) and silicon with an epitaxial layer of germanium (3-5K, i.e. zero within experimental error).

Considerable progress was made on the undercooling of melts that crystallize into polyhedral structures. The rebuilt 3m pyrex drop tube was used for solidification experiments on Ga-Mg-Zn droplets. This system is a known quasicrystal former. The aim was to investigate the degree of undercooling this melt can sustain, given that its

interfacial tension with polytetrahedral phases such as the quasicrystal, the quasicrystal approximants, and Frank-Kasper phases (such as the  $MgZn_2$  Laves phases) may be quite low. A large number of different sizes have been solidified. A detailed study of their microstructure is underway, including phase identification and quantitative characterization of the phase morphology. The ease of nucleation of the  $MgZn_2$  phase, which we observed in earlier work on undercooling of Al-Mg-Zn by different techniques, has been confirmed in this system.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 4/92 EXPIRATION: 3/95

PROJECT IDENTIFICATION: 962-25-07-07

RESPONSIBLE CENTER: JPL

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#### Presentations

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*Micro- and Macro-Segregation in Alloys Solidifying with Equiaxed Morphology*

PRINCIPAL INVESTIGATOR: Prof. Doru M. Stefanescu

University of Alabama, Tuscaloosa

CO-INVESTIGATORS:

L. Nastac

University of Alabama, Tuscaloosa

P. Curreri

NASA Marshall Space Flight Center (MSFC)

**TASK OBJECTIVE:**

The objective of this research is to extend the recently developed model for microsegregation from a closed system to an open system. This means that the new model should include the influence of (i) buoyancy-driven flow (thermosolutal convection), (ii) of convection caused by the relative motion of the liquid/solid interface, and (iii) of convection caused by density variation with change of phase, on both micro- and macro-segregation. The analysis will be conducted only for equiaxed dendritic and eutectic alloys.

Microgravity experimentation will be used to assess the relative value of the three factors affecting solute redistribution. The experimental work will be performed on the KC-135 aircraft for multi-directional solidification.

**TASK DESCRIPTION:**

The theoretical work will consist of the following tasks:

1. Develop a formulation to describe the rheology of the particular two-phase equiaxed systems of interest (dendritic and eutectic);
2. Develop an analytical solution for microsegregation for the case of an open system;
3. Develop a formulation for thermosolutal and shrinkage flow that includes description of both macro- and micro-segregation;
4. Couple the macro-transport to micro-transport through description of solidification kinetics;
5. Develop a numerical code to solve the above algorithm;
6. Evaluate the validity of the closed system microsegregation model for the case of low-g environment.

The open system model will be used in validation in conjunction with both high-g and low-g experiments. In addition, by turning off the shrinkage flow and/or the solid-liquid relative motion flow, the relative effects of these flows can be evaluated. The closed system model should come reasonably close to the low-g experiments. Validation of the models will be done through experimental measurement of solute concentration at the macro- and micro-scale level, and of the temperature distribution.

The equipment that will be used for the experiments is NASA's Isothermal Casting Furnace that has a temperature range of 100 to 1350 °C, and quenching rate capabilities of 1 to 50 °C/s. This furnace will be flown on the KC-135 aircraft. The samples will be melted before the low-g period of the flight, and solidified during the 25 s of low-g. Parallel experiments will be conducted on ground.

**TASK SIGNIFICANCE:**

At the micro scale level, the assumption that mass transport is purely diffusive is quite reasonable, and thus, a closed system may be a good approximation for certain alloy systems solidifying under terrestrial gravity. This assumption is even more valid when considering low-gravity (low-g) solidification. However, for most alloy

systems the influence of fluid flow caused by thermal and solutal convection cannot be ignored. During solidification of a multicomponent alloy, buoyancy-driven fluid flow occurs due to the temperature and concentration gradients. Even in upward directional solidification it is difficult to avoid horizontal temperature gradients since the container walls are not perfectly adiabatic. Accordingly, there is a need to further develop the model to address the open system case.

**PROGRESS DURING FY 1994:**

Funding for this project has been received in the fall of 1994. Consequently work has started only recently. To date a formulation to describe the rheology of the particular two-phase equiaxed systems of interest (dendritic and eutectic) has been developed.

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**STUDENTS FUNDED UNDER RESEARCH:****TASK INITIATION: 8/94    EXPIRATION: 8/96****PROJECT IDENTIFICATION: 962-25-08-34****NASA CONTRACT No.: Ncc8-059****RESPONSIBLE CENTER: MSFC**

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*The Impaction, Spreading, and Solidification of Partially Solidified Undercooled Droplets*

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PRINCIPAL INVESTIGATOR: Dr. Julian Szekely

Massachusetts Institute of Technology (MIT)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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**TASK OBJECTIVE:**

The purpose of this project is to examine the spreading of molten metal droplets and their solidification upon impacting onto a solid cooled surface.

**TASK DESCRIPTION:**

This project is aimed at studying the splatting and solidification behavior of undercooled and partially solidified metallic specimens. The project consists of both an experimental component and a computational and analytical component.

In the experimental arrangement a levitated and inductively melted metallic droplet will be allowed to fall in the Marshall drop tube, cool during flight, and then impinge on a chilled solid substrate. The flattened droplets will be collected and analyzed. The thermal management of the system will be so arranged that upon approaching the chilled substrate, the droplets will be undercooled in the range of 10-800 C, although, for some control experiments we will impinge superheated droplets or droplets close to their melting temperature. Furthermore, a number of experiments will be carried out, such that undercooled droplets will be made to impinge on the flat surface of the substrate.

However, perhaps the most novel feature of the investigation is that for the experiments, immediately prior to impingement of the droplet on the target, nucleation of the solid phase will be triggered by contact with one or more of many spikes raised above the substrate. The height of the spikes will be such that the specimen will be only partially solidified before impact.

Experiments to be run include impacts on a flat target by non-undercooled drops for comparison to previous work and undercooled drops to study the effect of the degree of undercooling on the stability (splashing tendency) and final microstructure of the drop. The study will also include the effect of controlled nucleation on the liquid-solid phase transformation by contact with a spike raised above the chill block. The occurrence of nucleation at a controlled time before impact will add a new dimension to the study of the competition between solidification and spreading of the droplet.

The theoretical work will include the development of a new set of equations to describe the behavior of the partially solidified droplet upon impact and the calculation of the rate of cooling of the specimen as it falls and the velocity field in the specimen as it falls. In addition, we will address issues of impingement and splatting.

**TASK SIGNIFICANCE:**

The problem of the spreading and solidification of molten metal droplets is of both fundamental and practical interest. The fundamental interest is provided by the fact that the simultaneous spreading and solidification of metal droplets is a generic problem in materials processing. The practical interest is associated with the immediate relevance of these phenomena to spray forming and the formation of coatings.

**PROGRESS DURING FY 1994:**

This research was initiated in June, 1994, and at the present time, we have done preliminary calculations assessing impact velocities and spreading times and have identified suitable candidate materials, which include aluminium, copper, zirconium, titanium, and nickel. In addition, we are planning to use two glass forming alloys developed in

## II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Prof. W. Johnson's laboratory at CalTech. We are hoping to initiate the first series of experimental runs at the Marshall Space Flight Center drop tower before the end of 1994. For the subsequent set of experimental series, we will initiate the nucleation of a solid phase just prior to impact and will assess the dropping characteristics of these materials.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-25-08-28

NASA CONTRACT No.: NAG8-1069

RESPONSIBLE CENTER: MSFC

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*Microporous Membrane and Foam Production by Solution Phase Separation: Effects of Microgravity and Normal Gravity Environments on Evolution of Phase Separated Structures*

PRINCIPAL INVESTIGATOR: Dr. John M. Torkelson

Northwestern University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

**TASK OBJECTIVE:**

The objective of this research is to develop a quantitative understanding of the microstructure formation in glassy, polymeric microporous membranes and foams produced via thermally induced phase separation. In particular, this study will delineate the role of gravity (resulting in buoyancy-driven flows resulting in macroscopic layering of phases) or the absence thereof in both the early-stage phase separation mechanisms, spinodal decomposition versus nucleation and growth, and the later effects of coarsening, by Ostwald ripening and/or hydrodynamic flow. Coarsening effects will be studied quantitatively by experimental determination of the growth in pore size over time as a function of quench depth, polymer molecular weight and concentration, surface tension, etc., in near-critical and off-critical point phase separation processes. Comparisons to expectations based on Ostwald ripening and hydrodynamic flow mechanisms of coarsening will be made. By developing a quantitative understanding of the thermodynamics and kinetics of polymer solution phase separation processes, both in terms of gravitational effects and the more ordinary effects of polymer/molecular weight/solvent/ concentration/quench temperature/quench time conditions, it may be possible to optimize microporous membrane and foam structure and performance.

**TASK DESCRIPTION:**

A microgravity environment will be simulated by use of isopycnic (iso-density) solutions of polystyrene in diethyl malonate, which upon thermally induced phase separation do not exhibit any macroscopic layering effects on the time frame of a week. Comparisons will be made to conventional, nonisopycnic polystyrene solutions. Early-stage spinodal decomposition effects will also be investigated by comparing the experimental temperature dependence of pore size to predictions from the linearized Cahn-Hilliard theory for spinodal decomposition. Measurements of the average pore size and pore-size distribution in membranes and foams produced by thermally induced phase separation will be characterized by scanning electron microscopy and mercury intrusion porosimetry. Comparisons of the growth rate in pore size observed from the three-dimensional membranes will be made to in situ measurements of phase separation and coarsening in polymer solutions by optical microscopy; the thin nature ( $\sim 10 \mu\text{m}$ ) of the solutions may yield two-dimensional coarsening effects.

**TASK SIGNIFICANCE:**

The effects of gravity in the production of microporous membranes and foams from phase separated polymer solutions will be studied quantitatively for the first time. By accessing microgravity conditions, it will be possible to study critically how the phase domain growth rate during coarsening compares to theories, including those related to the Ostwald ripening mechanism and the hydrodynamic flow mechanism of coarsening. From a scientific standpoint, by simulating microgravity conditions it will be possible to test for the first time the evolution of coarsened microstructure in polymer solutions over five to six decades in phase separation time and to determine how gravity-driven flows affect this evolution. From a technological standpoint, the microgravity environment using the isopycnic solution will allow determination of how space processing of microporous polymer membranes and foams may result in materials unobtainable or obtainable only for limited polymer species (where isopycnic solutions can be found) in a conventional terrestrial environment.

**PROGRESS DURING FY 1994:**

The phase separation of *isopycnic* polystyrene-diethyl malonate solutions has been studied by investigating the microstructure of polymer membranes. Polymer solutions underwent spinodal decomposition and coarsening via a thermally induced phase separation procedure, and supercritical CO<sub>2</sub> extraction was employed to remove solvent, resulting in microporous membranes. At relatively short coarsening times, the coarsening rate of the cell size can be expressed as a power law in time with the exponent increasing with increasing quench depth; for deep quenches, the growth rate has an exponent of 1/3 in agreement with the classic theories for coarsening by Ostwald ripening or coalescence. At longer coarsening times, there was a crossover to a much faster growth rate, yielding an exponent of 1.0 independent of phase separation temperature, consistent with expectations for the hydrodynamic flow mechanism of coarsening suggested by Siggia. This is the first experimental confirmation of the evolution of the coarsening mechanism from one mechanism with a growth rate consistent with Ostwald ripening or coalescence to a second mechanism with a faster growth rate. Comparisons were also made to coarsening observed in *nonisopycnic/low-viscosity* polystyrene-cyclohexane systems where strong gravitational effects dominate the phase separation process at relatively short times and crossover effects cannot be observed.

The effects of coarsening on microstructure formation in highly viscous polystyrene-cyclohexanol solutions and membranes made from them were also studied by scanning electron microscopy and mercury intrusion porosimetry. Using thermally induced phase separation and a freeze-drying technique, it was demonstrated that the polymer membrane microstructure can be tailored by controlling the quench route and coarsening time. For systems undergoing phase separation by spinodal decomposition, resulting in a well-interconnected, microporous structure with nearly uniform pore sizes, it was found that extending the phase separation time prior to freezing and solvent removal can result in a significant increase in pore or cell size which is highly dependent on both quench depth and coarsening time. The coarsening rate of the cell size can be expressed as a power law in time. For relatively deep quenches, the initial growth-rate exponent has a value of 1/3 in agreement with classical theories for coarsening by Ostwald ripening or coalescence, while for shallow quenches smaller exponents were observed, in agreement with studies involving isopycnic polystyrene-diethyl malonate systems. At longer coarsening times, a crossover to a much faster growth rate was observed yielding an exponent of 1.0, consistent with the expectations for the hydrodynamic flow mechanism of coarsening. This indicates that highly viscous, but nonisopycnic polymer solutions can, under certain circumstances, exhibit coarsening phenomena similar to that observed in an isopycnic, low viscosity polymer solution. Novel, complex microporous membrane structures with pore sizes of two characteristic length scales were also produced in this system using a two-step temperature jump process.

**STUDENTS FUNDED UNDER RESEARCH:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

**TASK INITIATION: 6/94 EXPIRATION: 6/96****PROJECT IDENTIFICATION: 962-21-08-23****NASA CONTRACT NO.: NAG8-1061****RESPONSIBLE CENTER: MSFC**

*Fundamentals of Mold-Free Casting Experimental and Computational Studies*

PRINCIPAL INVESTIGATOR: Prof. Grétar Tryggvason

University of Michigan

## CO-INVESTIGATORS:

S.L. Ceccio

University of Michigan

Dr. D. Jacqmin

NASA Lewis Research Center (LeRC)

## TASK OBJECTIVE:

- Provide the scientific knowledge necessary to operate a net-shape drop and spray casting facility by a computer designed and optimized process. In addition to its importance for earth based manufacturing, such knowledge will allow the operation of a space based facilities by computer programs developed on earth.
- Develop a detailed understanding of the fluid mechanics, heat transfer, and solidification of drops splatting on a solid surface or a layer of other drops, that can be used to predict the microstructure of solids formed by such depositions.

## TASK DESCRIPTION:

The project has both an experimental and a computational part. The experiment will consist of a facility that will allow drops of controlled size and velocity to be deposited on a flat substrata where they solidify. The numerical part will build on techniques developed under previous NASA support for accurate simulations of drop motion. By a careful comparison of the numerical and the experimental results, we expect to be able to identify the key physical aspects of the process that must be included in the numerical model so that it yields accurate results, yet is sufficiently fast to be of practical use. To scale-up the drops to allow more detailed observations of the process, we expect to eventually conduct the experiments in a low gravity environment where the drops can be made larger and the velocities smaller, while keeping the essential balance of physical effects the same as for the small drops that will be used in the actual production of artifacts.

## TASK SIGNIFICANCE:

Moldfree casting by precision controlled deposition of drops of molten metal is an emerging manufacturing process with considerable promise for rapid prototyping and the production of high quality, custom artifacts. A detailed understanding of the process, and the ability to predict the effect of the controlling parameters, is essential if the technique is to reach its full potential.

## PROGRESS DURING FY 1994:

The project has both an experimental and a computational part. The experiment will consist of a facility that will allow drops of controlled size and velocity to be deposited on a flat substrata where they solidify. Design of this facility is underway. The numerical part is building on techniques developed under previous NASA support for accurate simulations of drop motion. A computer program has been developed that models solidification together with two-phase (liquid-vapor) surface-tension induced flow. It is currently being tested by carrying out simulations of the solidification of a liquid with a free surface in a container.

## STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 7/94 EXPIRATION: 7/96

PROJECT IDENTIFICATION: 962-25-05-28

RESPONSIBLE CENTER: LeRC

*Electromagnetic Field Effects in Semiconductor Crystal Growth*

PRINCIPAL INVESTIGATOR: Dr. Martin P. Volz

NASA Marshall Space Flight Center (MSFC)

## CO-INVESTIGATORS:

K. Mazuruk

Hughes STX

D.A. Watring

NASA Marshall Space Flight Center (MSFC)

G.S. Dulikravich

Pennsylvania State University

## TASK OBJECTIVE:

The objectives of this investigation are the following:

1. To investigate the effects that combined electric and magnetic fields have on gravitationally-driven fluid flow processes during the bulk growth of selected semiconductor alloys.
2. To examine the criteria for the onset of thermal instability as a function of electric, magnetic, and gravitational field strength in electrically conducting liquids via theoretical analysis, computer calculations, and laboratory experiments.
3. To assess the possibility of using externally applied magnetic and electric fields to influence fluid motion in the melt, the rate of solid phase accrual, and the shape of the solid/liquid interface during solidification experiments both on earth and in a low gravity environment.

## TASK DESCRIPTION:

A series of specific tasks are planned to achieve the proposed objectives. A model growth cell will be developed which will allow for the passage of electric current through the melt simultaneous with the application of a magnetic field. In-situ temperature measurements will be made using gallium as a model material. Both zone-melting and vertical Bridgman thermal profiles will be applied and the onset of thermal fluctuations will be measured as a function of electric and magnetic field strength. Crystal growth experiments will then be made on GaInSb and CdTe under various electromagnetohydrodynamic conditions. Computer modelling of the above systems will be accomplished by using a three-dimensional mathematical model which takes into account both magnetohydrodynamic and electrohydrodynamic phenomena. The model predictions will be compared with the experimental results from the model growth cell.

## TASK SIGNIFICANCE:

Electromagnetic fields can interact with electrically conducting melts and substantially affect their fluid flow processes. Indeed, the intriguing possibility exists of using combined electric and magnetic fields to control fluid flow in the melt during the directional solidification of semiconductor alloys. The significance of this study is to gain a basic understanding of the possible interactions that combined electric and magnetic fields can have on fluid flow processes during semiconductor crystal growth.

## PROGRESS DURING FY 1994:

During the first three months of this investigation, construction was begun on a model growth cell. In addition, a cooperative agreement was established between MSFC and Pennsylvania State University so that the modelling effort can begin soon.



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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94    EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-21-08-24

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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*Coarsening in Solid-Liquid Mixtures*

PRINCIPAL INVESTIGATOR: Prof. Peter W. Voorhees

Northwestern University

## CO-INVESTIGATORS:

No Co-I's Assigned to this Task

## TASK OBJECTIVE:

The objective of this project is to plan and perform a microgravity experiment on Ostwald ripening in solid-liquid mixtures. This experiment will serve two primary purposes:

1. It will allow experiments to be performed which can be directly compared to heretofore untested theories for coarsening in systems with low volume fractions of solid.
2. It will eliminate conclusively convection of the liquid matrix and small-scale particle motion within the skeletal structure as possible sources for the disagreement observed between theory and experiment in the high volume fraction experiments.

## TASK DESCRIPTION:

Previous NASA sponsored work clearly showed that solid-liquid mixtures consisting of Sn-rich particles in a Pb-Sn eutectic liquid are ideal, and perhaps unique, systems in which to explore the dynamics of the Ostwald ripening process. The high coarsening rate in these systems permit accurate kinetic data to be obtained, and the thermophysical parameters necessary to make a comparison between theory and experiment are known. However, in a terrestrial environment experiments can be performed only at the relatively high volume fractions of solid where the presence of a solid skeletal structure prevents large-scale particle sedimentation. In these high volume fraction experiments, a comparison between theory and experiment shows that the solid-liquid mixtures are coarsening faster than predicted by an approximate theory for purely diffusional controlled Ostwald ripening. Thus, we are in the process of formulating a microgravity experiment. This experiment will serve two primary purposes: it will allow experiments to be performed which can be directly compared to heretofore untested theories for coarsening in systems with low volume fractions of solid, and it will eliminate conclusively convection of the liquid matrix and small-scale particle motion within the skeletal structure as possible sources for the disagreement observed between theory and experiment in the high volume fraction experiments.

## TASK SIGNIFICANCE:

The spaceflight experiment will produce data which, for the first time, can be compared directly to theory with no adjustable parameters. This data will address the long standing controversy over the dependence of the coarsening rate of a two-phase system of the volume fraction on coarsening phase.

The results from the spaceflight experiment will further our understanding of the processes responsible for microstructural development in a broad range of commercially important materials ranging from turbine blade alloys to liquid phase sintered materials.

## PROGRESS DURING FY 1994:

During this past year we began setting the science requirements for a spaceflight experiment. Specifically, we have examined the effects of g-jitter on the motion of a solid particle in a fluid, examined the effects of long-term storage at elevated temperatures on the microstructure of previously coarsened Pb-Sn samples, and prepared the preliminary science requirements document. Our theoretical investigation on the effects of g-jitter showed that g-jitter of the relatively small amplitude and frequencies characteristic of routine crew movement on the space shuttle can induce significant motion of Pb solid particles in a Pb-Sn eutectic liquid. Thus, we were able to determine that the

spaceflight experiment must be performed during crew sleep periods. In addition, the long-time annealing studies showed that either following or preceding the spaceflight experiment the samples cannot be exposed to temperatures in excess of 30C. Finally, we have been interacting with engineers at NASA Lewis in the design of a furnace which can be used in a microgravity coarsening experiment. We have also continued our ground-based research. We have examined experimentally the dynamics of transient coarsening in solid-liquid systems, and are in the process of developing an automated serial sectioning machine. The transient coarsening experiments showed clearly that both the spatial arrangement of the particles and the form of the initial particle size distribution can have a major effect on the dynamics of the coarsening process at short times. However, as coarsening of the microstructure proceeds, the system approaches the classical steady-state coarsening process. The automated serial sectioning machine will be used to analyze the results of the spaceflight experiment as well as to explore the three-dimensional morphology of the solid-liquid mixtures produced both on the ground and in space.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/92 EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-25-05-10

NASA CONTRACT No.:NAG3-1417

RESPONSIBLE CENTER: LeRC

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*Containerless Liquid Phase Processing of Ceramic Materials*

PRINCIPAL INVESTIGATOR: Dr. Richard J. Weber

Containerless Research, Inc.

CO-INVESTIGATORS:

P.C. Nordine

Containerless Research, Inc.

**TASK OBJECTIVE:**

This research uses the control of chemistry and nucleation achieved by containerless liquid-phase processing to study non-equilibrium phase formation and crystal growth. The work is intended to advance the basic understanding of the high temperature chemistry of hard, refractory oxide and boride ceramics. Borides are of fundamental interest; they are a unique class of compounds which form highly covalent, complex crystalline structures.

Ground-based containerless experiments will enable non-equilibrium phase formation phenomena to be identified. This will allow candidate materials for more detailed investigation to be selected. Subsequent low gravity experiments will provide the high degree of control over molten specimens required for detailed studies and analyses of the liquids as well as crystal growth kinetics and solid-liquid phase relationships.

**TASK DESCRIPTION:**

High temperature liquid-phase processing is achieved by aero- acoustic and aerodynamic levitation in combination with continuous wave CO<sub>2</sub> laser beam heating. Levitated materials are viewed by optical pyrometers and video cameras. Materials are being examined by optical and scanning electron microscopy, X-ray diffraction, nuclear magnetic resonance, Raman spectroscopy, and laser fluorescence measurements.

The effects of melt chemistry, temperature and process variables, including gravity-driven convection on solidification kinetics, metastable phase formation, and epitaxial growth onto isostructural seed crystals from undercooled melts is being investigated.

**TASK SIGNIFICANCE:**

The work provides insights into subtle chemical and transport effects on the solidification of complex oxide and boride melts. The investigation explores the limits of ground-based methods and identifies systems which require the additional control of transport which may be possible in low gravity.

**PROGRESS DURING FY 1994:**

The investigations have so far concentrated on work with oxide- based melts. Materials selected were calcia-gallia (C-G) mixtures (in collaboration with Professor D.E. Day and Dr. C.S. Ray from University of Missouri-Rolla), aluminum oxide, yttrium-barium-copper oxide ceramic superconductor materials, and rare-earth borides. Most of the results have been published or presented:

1. Measured effects of solid contact on C-G glass formation characteristics
2. Investigated aluminum oxide structure-properties as a function of p(O<sub>2</sub>)
3. Established control of YBaCuO phase relationships via oxygen pressure
4. Conducted containerless melting of boride materials
5. Conducted collaborative investigations with scientists at Argonne National Laboratory, Jet Propulsion Laboratory, University of Wisconsin, Madison, and Vanderbilt University.

The present work has achieved a high degree of control of melt chemistry, nucleation, and the gas environment in ground-based containerless experiments on high temperature melts. Examples where increased control is needed are the following:

1. Glass formation - Stirring enhanced crystallization rates by transporting nuclei through melt.
2. Aluminum oxide - temperature gradients limited the ability to process and investigate liquids in uniform thermodynamic states.
3. YBaCuO material - segregation of  $Y_2O_3$  particles limited control of the product microstructure.

Melt properties depend on composition and temperature and the composition itself depends also on temperature under fixed ambient conditions. Microgravity provides access to more uniform melt temperatures and compositions as well as control of fluid motion needed for transport property measurements. Measurements of melt properties are a key to understanding solidification behavior in ceramic systems, large changes in viscosity result from the response of liquid structure to temperature and composition changes. Experiments on the NASA KC-135 are planned in a collaboration with a Canadian research group.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/92 EXPIRATION: 5/95

PROJECT IDENTIFICATION: 963-26-07-03

RESPONSIBLE CENTER: JPL

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### *BSO/BTO Identification of Gravity Related Effects on Crystal Growth, Segregation, and Defect Formation*

PRINCIPAL INVESTIGATOR: Prof. August F. Witt

Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

#### TASK OBJECTIVE:

Knowledge gained from space experiments and the related ground-based program is expected to advance the science base for crystal growth of oxides and thus to narrow the still existing gap between theory and experiment. More specifically, the proposed program provides an approach to the deconvolution of the effects of largely uncontrolled, complex processing variables on crystal growth and segregation, leading to the identification of growth conditions by which device specific property requirements in oxides can be approached. Such growth conditions are expected to be realizable in a modified Bridgman growth geometry, the subject of development in the ground-based support program.

#### TASK DESCRIPTION:

The on-going research program on growth and characterization of BSO places focus on a class of materials (optical and opto-electronic) with theoretical properties that are outstanding, but which have so far failed to reach their potential primarily because of our inability to adequately control during growth their stoichiometry, incorporation of functional minority constituents, crystal defect formation and confinement related contamination. Theoretical considerations indicate that the majority of existing growth deficiencies, which are responsible for our inability to produce viable device structures, are directly or indirectly related to gravitational effects. Thus it appears that an assessment of the true potential of selenites, which exhibit outstanding piezoelectric properties and exceptionally light optical rotative activity in device application, can at this time best be made on material obtained from controlled growth experiments in a reduced gravity environment. Under such conditions convective interference, otherwise unavoidable, is projected to be substantially suppressed and defect structures resulting during growth are expected to approach equilibrium values. Magnetic melt stabilization, found effective in growth of semiconductors, is ineffective in oxide systems.

#### TASK SIGNIFICANCE:

The research to date has focused on (a) the development of BSO single crystal growth capability by the Czochralski technique, (b) the characterization of growth and defect formation in BSO, and (c) the development of a Bridgman-type growth configuration for BSO that permits enhanced heat transfer control under quantifiable thermal boundary conditions and which is functionally compatible with NASA -generated existing hardware. The establishment of quantifiable growth conditions and the *in situ* growth characterization capability by means of current induced interface demarcation are considered essential in efforts directed at assessing the potential of reduced gravity environment for crystal growth research and development since the customary empirical approach is considered prohibitive in space experimentation. The approach taken is in addition also considered a key element in efforts to establish advanced crystal growth capabilities on earth, a prerequisite for the meaningful assessment of the potential of selenites in device applications.

#### PROGRESS DURING FY 1994:

##### Crystal Growth of BSO

A heat pipe-based Czochralski system, providing for a controlled ambient atmosphere and current induced growth interface demarcation, has been established and used extensively to grow material from which growth and segregation effects in conventional operation are being determined.

Characterization

In view of extensive experience in optical characterization gained from NASA-sponsored research of III-V and II-VI compound semiconductors, existing facilities for optical transmission microscopy and related computational image processing and analysis were modified to permit the characterization of oxides.

To determine the basic growth features of BSO, Ga-doped as well as undoped crystals using charges ranging from 50-70g were grown and analyzed. It was found that (1) rotational striations are absent in crystals grown with the heat pipe based system; (2) non-rotational striations, highly periodic in nature and exhibiting two distinctly different frequencies, are present in both the core and off-core regions; (3) the frequency of striation formation appears to be a weak function of the aspect ratio of the charge, independent of the rate of seed rotation and not noticeably dependent on the rate of crystal pulling; (4) the primary crystal deficiencies are gaseous inclusions; their appearance is a function of both the rate of crystal rotation and the rate of crystal pulling; (5) generation of dislocations is found to be predominantly related to inclusion of "gas bubbles"; (6) dislocations have been made "visible" through decoration by annealing in a reducing atmosphere; and (7) current induced interface demarcation has been successful - its formation can be attributed to Joule heating rather than a Peltier effect.

Design of Bridgman Facility

A heat pipe based Bridgman facility has been designed and constructed. The system is equipped for interface demarcation and will provide growth rate as well as thermal gradient stabilization. It is currently being tested for performance characteristics using a "graphite charge" and will subsequently be used to establish growth conditions for BSO in vertical configuration.

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STUDENTS FUNDED UNDER RESEARCH:

BS Students:	0	BS Degrees:	0
MS Students:	0	MS Degrees:	0
PhD Students:	2	PhD Degrees:	1

TASK INITIATION: 1/93 EXPIRATION: 1/96

PROJECT IDENTIFICATION: 962-21-08-19

NASA CONTRACT NO.: NAG8-949

RESPONSIBLE CENTER: MSFC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Microwave Furnace Development for Microgravity Materials Processing*

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PRINCIPAL INVESTIGATOR: Dr. Martin B. Barmatz

Jet Propulsion Laboratory (JPL)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

The objective of this Advanced Technology Development (ATD) proposal is to build a highly efficient, cold-wall, direct-heating microwave furnace that will permit fast heating of microwave-absorbing materials and quick cooling of these materials when the microwaves are removed. The task objectives will include 1) development of advanced energy saving methods for (a) continuous tracking of the cavity resonant mode during processing, (b) continuous matching of source and cavity impedance during processing, 2) design, fabrication and evaluation of various cavity geometries and excitation techniques, and 3) modeling of the microwave heating process for various sample shapes and sizes.

#### **TASK DESCRIPTION:**

In the future, many materials processing studies in space will require specialized furnaces, particularly at high temperatures. Requirements for these furnaces include fast, controlled sample heating, high precision sample temperature control, and fast, controlled sample cooling. Furthermore, there are constraints that a microgravity furnace should meet; i.e., they should be small, light weight, and very energy efficient. Properly designed microwave furnaces can deliver most of the generated energy directly to the material being processed, thus leading to improved energy efficiency. The design requirements for energy efficiency include critical coupling of microwave energy from source to cavity, high electrically conducting cavity walls compared to sample conductivity, and continuous tracking of the microwave resonance during processing. Furthermore, microwaves are able to directly heat the interior of the materials, which differs from conventional heating methods, where only the sample surface is directly heated. In some cases, this microwave volumetric heating ability can lead to more uniform processing, while in other cases it may lead to an internal temperature profile with the highest temperature in the center of the material. The furnace designs developed during this ATD task will allow unique microwave heating properties as well as precision sample temperature control capabilities to be applied to microgravity processing. By the proper choice of sample shapes, such as spherical or cylindrical, one can also theoretically predict the unique heating behavior of the sample interior during processing.

#### **TASK SIGNIFICANCE:**

The unique microwave volumetric heating capabilities will permit new and novel scientific studies to be performed in microgravity. At this time, scientific and industrial communities are re-visiting the use of microwaves as a means to process materials on the ground, particularly at high temperatures. Many new materials processing techniques using the unique capabilities of microwaves are now being evaluated and the technology associated with this ATD should play an important dual-use role in developing improved commercial products.

#### **PROGRESS DURING FY 1994:**

During the last year, we have (a) developed a new vibration technique for tracking the microwave mode resonance in a 1 Kw magnetron facility using a motor plunger, (b) extended the above vibration technique to track the impedance match between the microwave source and processing cavity in a 1 Kw magnetron facility using a motor controlled stub tuner, and (c) developed a microwave heating model for determining the steady state temperature profile in a cylindrical sample in a cylindrical cavity.



## II. MSAD Program Tasks — Advanced Technology Development

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/91    EXPIRATION: 9/94

PROJECT IDENTIFICATION: 92-1

RESPONSIBLE CENTER: JPL

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Stereo Imaging Velocimetry*

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PRINCIPAL INVESTIGATOR: Dr. Mark Bethea

NASA Lewis Research Center (LeRC)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

The objectives of this project are to develop a stereo imaging velocimeter that will:

1. Measure quantitatively and qualitatively velocities up to 10.0 cm/sec with an accuracy of 2.0% of full-field for 150-micron seed particles in a 2.0-inch field of view.
2. Have streamlined data processing which processes 100 time steps of consecutive stereo images to obtain 3D velocity fields in less than ten minutes.
3. Be able to track at least 100 particle pairs per frame.
4. Require minimal apriori assumptions about the flow.
5. Initiate tracking and matching out automatically.

#### **TASK DESCRIPTION:**

The approach to successful implementation is to develop robust and efficient 3D camera calibration, edge finding/centroid determination, overlap decomposition, particle tracking, and stereo matching algorithms that will be used in the Stereo Imaging Velocimetry system. These five tasks are the basis for the velocimeter and will determine its final processing speed and accuracy. We will then test the prototype for accuracy on particles with known trajectories. A user interface for both the front-end and post-processing will be created. The velocimeter will be tested on real fluids experiments. This testbed experiment will be done with a water tunnel experiment that is currently under development. Using the water tunnel experiment, we can set the flow rate and compare the results with the SIV system results.

#### **TASK SIGNIFICANCE:**

Stereo Imaging Velocimetry will permit the collection of quantitative and qualitative, three-dimensional flow data from any optically transparent fluid which can be seeded with tracer particles. This includes such diverse experiments as the study of multiphase flows, bubble nucleation and migration, pool combustion, non-contact measurements, and crystal growth -- all of which are part of NASA's Microgravity science program.

#### **PROGRESS DURING FY 1994:**

A neural network approach to particle tracking has been invented, coded, and tested. The results show a substantial improvement over our existing particle tracking scheme. This work is significant in that we use a globally optimum neural network instead of a locally optimum one. This increases our particle tracking yield by as much as 10%.

A 3D camera calibration technique has been developed and tested for the SIV system. We are now able to take two 2D calibration (orthogonal views) and extract 3D information from then to produce a robust 3D camera calibration routine capable of calibrating our fluid volume to within 0.17% of full-field.

## II. MSAD Program Tasks — Advanced Technology Development

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The Neural Network implementation of the overlapping particles reported in FY 93 has been combined with our edge finding-centroid processing algorithm to form one robust algorithm.

The 3D camera calibration technique, overlap decomposition with edge-centroid determination, along with particle tracking and stereo matching are currently being combined in one SIV algorithm. This will produce our SIV software system package and accuracy and speed will be analyzed.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students:	1	BS Degrees:	0
MS Students:	1	MS Degrees:	3
PhD Students:	2	PhD Degrees:	0

TASK INITIATION: 10/91    EXPIRATION: 9/95

PROJECT IDENTIFICATION: 963-70-0C-00

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Real-Time X-Ray Microscopy for Solidification Processing*

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PRINCIPAL INVESTIGATOR: Dr. Peter Curren

NASA Marshall Space Flight Center (MSFC)

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CO-INVESTIGATORS:

W. Kaukler

University of Alabama, Huntsville (UAH)

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#### **TASK OBJECTIVE:**

The objective of this ATD consists in the development of an X-ray Transmission Microscope (XTM) for the in-situ and real-time observation of interfacial processes in metallic systems during freezing or solid-solid transformation.

The XTM should have the following capabilities:

1. provide a resolution for specimen features of 10-100 nm;
2. at solidification rates of 0.1 to 20 mm/sec;
3. temperatures up to 1100 C with temperature gradients up to 50 C/cm;
4. with contrast sensitivities sufficient to detect 2-5% difference in absorptance;
5. offer 1, 2 and 4 in exposure times of a few seconds; and
6. permit recording of stereo pairs for depth information.

#### **TASK DESCRIPTION:**

The purpose of this ATD is the development of a high resolution x-ray either during freezing or solid-solid transformations. The X-ray Transmission microscope to view, in-situ and in real time, interfacial processes in metallic systems Microscope (XTM) will operate in the hard x-ray range (10 to 100 keV) and achieve magnification through projection.

#### **TASK SIGNIFICANCE:**

Physical processes which occur at, or near, interphase boundaries during solidification, or other phase transformations, play a major role in the determination of many of the technologically important properties of solids. To-date, interfacial morphologies and particle-interface interactions in the respective metallic, optically opaque systems have been deduced from post-process metallographic analyses of specimens. Thus, little information is obtained about the detailed dynamics of the processes. These investigations have been considerably augmented by real time observations of transparent materials; yet, since some of the interfacial and transport properties of these materials differ greatly from those of metals and semiconductors the results are not necessarily representative of these opaque systems.

#### **PROGRESS DURING FY 1994:**

Procurement was achieved of a system which is of a capability of the order to that which we utilized for the proof of concept studies. It basically consists of a Microfocus X-ray source, a X-ray image converter, and a CCD detector. The sub-micron X-ray source is a relatively new technology and will not be improved upon during this ATD. The detector / converter system of the unit is not optimized for real time analysis and will be improved as part of the ATD effort.

A first principles adsorption model approach was initiated for determining the useful contrast from a sample. Using standard materials data and published X-ray spectra, the degree of absorption was calculated of the rays as they pass through the inhomogeneous sample. Resolution limits established by the converter / camera combination were introduced via Modulation Transfer Function, MTF, data for the X-ray image intensifier. This latter area of modeling will require much more work to predict which conditions and apparatus will be optimum. The effort to date provides useful information about the probability of observing important microstructural features and how high a contrast the feature may have under ideal conditions.

## II. MSAD Program Tasks — Advanced Technology Development

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Several X-ray conversion technologies have been surveyed for applicability to this project. Due to the real-time requirements, film is not suitable for use in this project except to record images of stationary specimens. Only technologies that offer instant or real-time images will be considered.

Evaluation of new CCD X-ray Converter and camera technology has been performed. This technology uses radiation hardened CCDs as a direct conversion, hard x-ray detector for medical purposes. This competitive technology may offer a superior means of seeing the features microstructural features of most scientific interest with increasing resolution and potentially higher gray level sensitivity properly called dynamic range.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/94    EXPIRATION: 10/97

PROJECT IDENTIFICATION:    963-70-04

NASA CONTRACT No.:    In-house

RESPONSIBLE CENTER: MSFC

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### BIBLIOGRAPHIC CITATIONS FOR FY 1994:

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Microgravity Combustion Diagnostics*

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PRINCIPAL INVESTIGATOR: Paul S. Greenberg

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CO-INVESTIGATORS:

Dr. D.W. Griffin

NASA Lewis Research Center (LeRC)

Dr. R.L. Vander Wal

NYMA, Inc.

Dr. K.J. Weiland

NASA Lewis Research Center (LeRC)

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#### **TASK OBJECTIVE:**

Currently available diagnostic instrumentation for achieving these objectives has been extremely limited, consisting primarily of conventional film-based imaging systems and intrusive temperature and velocity probes, such as thermocouples and hot wire anemometers. This situation has arisen primarily because of the unique and severe operational constraints which are inherent in the conduct of reduced-gravity experimentation. It is the recognition of this pressing need to provide diagnostic systems of greater sophistication that has motivated the existence of this particular development program.

#### **TASK DESCRIPTION:**

For a variety of reasons, predominant emphasis has been placed on the development of optical diagnostic techniques. Principal among these is the relative fragility of the physics and chemistry of reduced-gravity systems relative to their 1-g counterparts. The action of buoyancy-induced convection is vigorous when compared with the dominant mechanisms associated with reduced-gravity phenomena, such as surface tension and thermal and concentration driven diffusion processes. The essentially nonperturbative nature of optical measurement techniques is therefore extremely appropriate in this context.

Optical measurement techniques are, in general, well-suited to the acquisition of multidimensional data fields (e.g., two- and three-dimensional imaging). This is an important consideration in the present state of understanding of microgravity science, since a clearer understanding of basic phenomenology, including the verification of fundamental length and time scales and dominating physical mechanisms is still being developed.

#### **TASK SIGNIFICANCE:**

The success in achieving a significant scientific return from existing and proposed microgravity fluid physics and combustion science experiments depends substantially on the availability of diagnostic systems for the collection of the required scientific data.

#### **PROGRESS DURING FY 1994:**

In the area of full-field infrared emission spectroscopy, calibration of the IR sensitive staring array camera has been conducted. This was accomplished through the use of a blackbody radiation source and several infrared bandpass filters. An end-to-end calibration is required in this case because of the varying response of the detector array and the transmission characteristics of the lens and bandpass filters. All of these elements exhibit behavior that is wavelength dependent; the detector response may exhibit nonlinearities with respect to absolute intensity as well. The response of the detector array has been observed to be nearly proportional to blackbody intensity when narrow band filters are employed. As a first step in exploiting this calibration, images of radiating thin filament fibers suspended in jet flames have been obtained. The resulting data will be compared to thermocouple measurements of the hot gases above the flame that were obtained simultaneously.

A preliminary literature search was made investigating the inverse problem wherein infrared emission measurements are inverted to provide temperatures and concentrations of selected species. The present search focused on references comparing modelling efforts and experimental data for an experimentally tractable (soot-free, relatively simple chemistry, straightforward geometry) combustion system. Published infrared spectra of some laboratory flames, such as a Bunsen-type natural gas flame, have also been found.

## II. MSAD Program Tasks — Advanced Technology Development

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Independent of this ATD, a Phase II SBIR contract was signed with SSG, Inc. for the development and subsequent delivery of an infrared imaging spectrometer capable of supplying spatially and spectrally resolved images. Two design reviews for the instrument were held to finalize design requirements and specifications. As part of this effort, an estimate of the radiation levels expected from microgravity flames was required to assess the required instrument sensitivity. No microgravity data was found to exist, so published data from measurements conducted in the laboratory on laminar and turbulent gas jet flames was used. In addition, the DARTFire Co-Investigator provided information on her calculations of the radiation levels expected from burning PMMA in microgravity.

Efforts supporting two-dimensional species and temperature measurements were initiated with the completion of the procurement process for the titanium:sapphire laser. The laser, built by Continuum, Inc., was installed into a new laboratory room in the Space Experiments Laboratory. Through frequency doubling, tripling, and mixing of the titanium:sapphire fundamental output, light in the blue and ultraviolet has been successfully generated. These lines cover the wavelength regions of 431 nanometers and 308 nanometers, providing the ability to perform laser-induced fluorescence measurements of the CH and OH radicals, respectively. This laser system is currently being characterized as to its operational capabilities. Optics and other support equipment (burners, chamber, etc.) to perform Rayleigh scattering, laser-induced fluorescence, and other optical diagnostics have been obtained and are presently being assembled.

Characterization of this new device began by looking at water, which is optically active throughout the fundamental wavelengths of the laser and allowed familiarization of the laser system without the worry of frequency conversion. A photoacoustic cell and associated electronics were constructed. Excitation of various absorption bands of water at room temperature can be detected, thus serving as a frequency marker to reference the laser tuning process. Synchronization of the laser pulses, data collection systems, and wavelength tuning has also been implemented.

Next, the frequency doubling capabilities of the laser were characterized. Several millijoules of light could be produced at 390 nm, which is in the range of one of the CH radical electronic transition. The laser was set up to automatically scan the frequency conversion crystal angle as the wavelength is scanned. The scan rate for auto-tracking was determined to be at least 0.1 nm/sec. The tuning range of the laser when optimized at a particular wavelength is greater than 60 nm, although the auto-tracking alignment must be slightly realigned for good tracking at roughly 20 nm intervals.

A Bunsen-type methane/air flame was set up and a point, laser-induced fluorescence signal of the CH radical present in the inner cone of the flame was obtained with the use of a monochromator and photomultiplier tube detection system. The peaks were assigned using published line positions. From the difference in the actual versus measured wavelength of about 2.32 nm, a wavelength calibration of the laser can be made.

Exciplex thermometry experiments are being performed utilizing a small pulsed nitrogen laser operating at 337 nanometers. The objective is to utilize this method to measure the temperature of a vaporizing droplet, and extend the technique to a burning droplet. Exciplex thermometry utilizes the ratio of fluorescent intensities of an excited state monomer and exciplex compound (formed through the interaction of an excited state monomer and ground state partner). By referencing this ratio of fluorescence intensities to calibration measurements in which the intensity ratio is measured at known temperatures, the temperature can thus be determined. This technique has been applied to measure the volume average temperature of a fiber suspended droplet in an inert atmosphere in response to heating by a hot wire coil surrounding the droplet. The system in this case is hexadecane doped with PYPYP. Application of exciplex thermometry to burning droplets remains a challenge as the fluorescence from the monomer and exciplex are quenched at different rates in the presence of oxygen. Current efforts are aimed at addressing this issue.

Point-wise and imaging velocity measurements are also in progress. Addressing the former, compact, solid-state laser doppler velocimeter modules have been obtained on loan to supplement the module being completed under contract. This module is implemented for coaxial backscatter measurements, utilizing common optics for simultaneous transmission and detection. This simplifies the experimental geometry, and eliminates the requirement for precise alignment of separate transmission and collection sample volumes. Acceptance tests of the optics module were performed during September of 1993 at EG&G's facility in Montreal. The results of these tests indicate that the unit performed as per specification. A comparative basis for the resulting performance was afforded

## II. MSAD Program Tasks — Advanced Technology Development

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by the availability of a pair of similar units presently in use in our laboratory. These units were loaned from the David Taylor Institute of the Naval Research Laboratory. Both the EG&G and NRL units employ diode laser sources operating at 780 nanometers, delivering approximately 15 milliwatts at the sample volume. Both units also employ DC coupled, avalanche photodiodes operating in the sub-geiger module (i.e. analog detection as opposed to photon counting). For comparable optical power present at the sample volume, the EG&G module affords an improved detection sensitivity by roughly a factor of four. A two-fold improvement is evident directly from the indicated data, whereas the additional factor of two results from F# considerations; both modules employ a collection aperture of 50 millimeters, but the EG&G module has a working distance which is 1.4 times as long. Also visible in the data is the considerable improvement in the spatial definition afforded by the EG&G module. The radial profile of the sample volume is very close to the predicted value of 100 microns, and much more closely resembles the  $1/e^2$  profile associated with the source. The NRL unit, in contrast, has inferior beam shaping optics, resulting in a series of unwanted "wings" which contribute to the overall scattered signal.

Upon receipt of the EG&G optics module at LeRC these tests were repeated. Unfortunately, two anomalies in the performance of the system were observed which were not seen previously. The first corresponds to a misalignment between the transmitted and detected sample volumes. Data was collected to indicate the output of the avalanche photodiode detector as a partially reflective target was translated along the optical axis. It is seen from the resulting plot that the detection sensitivity peaks approximately 20 millimeters behind the sample volume as defined by the transmitter. Because the detection optics operate at a conjugate ratio on the order of 10:1, this is indicative of a misalignment of the detector by roughly two millimeters. This distance was later revealed to correspond to a shift in the distance between the entrance window and sensor surface that had been altered in the final version of the detector. This change had evidently not been indicated in the final configuration drawings, and hence had become inadvertently misaligned in the final cleaning, purging, and sealing of the module which occurred prior to shipment. The second anomaly relates to 50 KHz bleed-through from the detector high voltage power supply, and was traced to a leaky by-pass capacitor. The module has subsequently been returned to EG&G, where these problems are presently being corrected.

In the interim, the NRL units continue serve as a basis for further system development. A TSI-IFA 750 burst spectral processor has been obtained on long-term loan from the Propulsion Systems Division here at the Laboratory, and is presently being utilized for assessing the performance of particle seeders suitable for reduced gravity applications. Conventional seeders are predicated on fluidized beds or sedimentary reservoirs, both configurations being inappropriate for reduced gravity applications. As an initial step in establishing familiarity with this combination of optics module and processor, velocity profiles were acquired from a non-reacting jet seeded with a water vapor nebulizer. Turbulence intensities in excess of 30% were calculated from 1K sample pdf's, and have the expected spatial profile associated with laminar-to-turbulent transitions of this type. Also obtained is the data rate associated with each sample point. These rates peak at roughly 9.6K velocity estimates per second, falling short of the capacity of the processor by more than 10:1. This behavior is attributed to the polydispersity of the seeder; previous measurements indicate that more than half of the droplets produced are well below 0.1 micron in diameter, yielding visibilities which are below the measurement threshold of the combined instrument. When operating closer to the throughput capacity of the processor (120K estimates/sec), turbulence estimates derived from similar pdf's should be obtainable in 10 - 20 milliseconds/point. This increase in arrival rate (i.e. validated and processed) of resolvable particles will be sought from subsequent seeder designs, which are based on the entrainment of near monodisperse particles in a fully turbulent enclosure. The throughput that is ultimately achieved from such a system will provide an upper bound for the rate at which multi-point spatial scans may be performed.

In the area of Particle Image Velocimetry, a specially configured, pulsed Nd:YAG laser was received. This laser was designed to emit a 4 pulse burst every second; shots in the burst are repeated at a rate of 1KHz. It also can produce single pulses with a 30Hz repetition rate, although the energy produced is much less. The output is frequency doubled to 532 nanometers via an internal doubling crystal. This laser uses a recently developed folded-resonator configuration, resulting in an extremely compact optics package. Although some "tweaking" of this system was required by the manufacturer, subsequent acceptance testing of this unit has validated that the specified performance has indeed been achieved. In addition to the rugged, compact packaging of the optical head, this laser system is configured so as to be compatible with operation in either the NASA Learjet or KC-135 aircraft.



## II. MSAD Program Tasks — Advanced Technology Development

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Following receipt of the laser, a cyclonic insertion fluidized bed seed chamber was designed and fabricated. This seeder is intended for use with gas jets which are identical to those in use by investigators currently funded under the MSAD program. Clear acrylic was used as the material so that the flow structure in the interior could be viewed. Testing revealed that the injection velocities were too low to entrain sufficient numbers of particles due to the diameter of the inlet port and the relatively small mass flow rates employed in these experiments. Additionally, the flow tended to "tunnel" through the particle bed which further precluded entrainment. Based on these results, a new design was developed, with the goal of developing fully-turbulent flow inside the seeder plenum. The diameter of the burner, which forms the outlet of the chamber is 1.65 mm. With an inlet diameter of approximately 0.1 mm, a Reynolds number of roughly 2000 inside the seeder was achieved. This design was tested using a new type of seed particle known as "Microspherical Feathers," manufactured by Osaka Gas and discovered by Paul Greenberg while working on the LDV portion of this project previously described. These particles are essentially hollow spheres of silicon dioxide, and are extremely monodisperse. They possess extremely large scattering cross sections and small effective hydrodynamic diameters, both properties being advantageous for this application. Initial testing showed that the particle entrainment was excellent and velocity vectors were determined using images captured both live and from a pre-recorded sequence recorded in Sony BetaCam format. To simplify the experimental configuration, the illumination for these tests was provided by a CW argon-ion laser. The data reduction algorithm being utilized was developed by Dr. M. P. Wernet of the Instrumentation and Control Technology Division of LeRC, and has been previously described.

Numerical aspects of modeling for soot formation and radiation heat transfer have been completed. Soot transport equations, the energy equation, and solutions for the radiative transport equation (RTE) have been incorporated into a parabolic solver code for nonpremixed turbulent free-shear layer flows. The RTE can be solved by the YIX method or via the method of spherical harmonics (PN). The former is considerably more accurate and suitable for inhomogeneous media. It is disadvantageous, however, due to the requirement for long computation times. The latter is computationally more expedient, but has been observed to exhibit numerical instabilities in the optically thin limit. Iterations are then required between the energy equation of these solutions. Reasonable agreement has been found between predicted and measured soot volume fraction and temperature data. The final part of modeling to be completed is an accurate and efficient way for spectrally integrating the spectral radiative fluxes and properties.

Thermophoretic probe sampling measurements and subsequent TEM analysis for soot particle size and morphology have been completed in the laboratory on propane and ethylene diffusion flames. Full-field laser-light extinction measurements have also been successfully demonstrated. The tomographic inversion of the data to provide spatially resolved mass fractions has also been demonstrated. These results are presently being assembled for publication. A package has been constructed to perform both of these measurements in the LeRC 2.2 sec. drop tower. Data elucidating primary particle size distributions for both propane and ethylene diffusion flames under both normal and reduced gravity conditions has been successfully obtained and is being submitted for publication. Reduced gravity measurements of soot volume fraction were not, however, completed before the shutdown of the 2.2 second tower and hence will resume pending the availability of this facility. In the interim, other methods are being pursued in the laboratory, including interlacing fine, rapid response thermocouples between the probe grids, simultaneous absorption/pyrometry measurements, and possibly flow-field mapping via laser doppler velocimetry.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/92    EXPIRATION: 9/96

PROJECT IDENTIFICATION: 93-2

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Small, Stable, Rugged Microgravity Accelerometer*

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PRINCIPAL INVESTIGATOR: Frank T. Hartley

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#### CO-INVESTIGATORS:

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#### TASK OBJECTIVE:

The objective of this ATD task is to build a novel micromachined accelerometer that is capable of measuring accelerations from  $10^{-2}g$  to  $10^{-8}g$  with a better than  $10^{-8}g$  accuracy ( $<10^{-9}g/\sqrt{Hz}$ ) for the frequencies from  $10^{-4}$  to 20 Hz. The device should have low temperature sensitivity and have a build-in calibration. The accelerometer must withstand the launch environment. The task objectives include: 1) development of micromachined flexures; 2) development of an electronic parking mechanism; 3) development of active controls for the accelerometer, and 4) development of electronics for data acquisition and control.

#### TASK DESCRIPTION:

The most important parameters are independent measurements of all three spatial components of acceleration, high accuracy, and low-frequency measurements. The sensitivity, accuracy and frequency domain are determined by the different acceleration sources that are found aboard the Space Shuttle and other spacecraft. The lowest frequency is determined by the low Earth orbit period. The "as designed" device should be able to measure accelerations at frequencies lower than  $10^{-4}Hz$  – 1/90 minutes, but the accuracy will decrease. Inertial navigation requires precise positioning in space as well as the orientation tracking (sets of six or more accelerometers working as a three-dimensional gradiometer). Seismology applications require high sensitivity in the presence of a constant acceleration (g). All of these applications are possible with a flexure suspension and electrostatic actuation design of the accelerometer. The flexure permits precise measurements of very small accelerations in the presence of a large cross-axial acceleration. In other words, the accelerometer can measure the components of the acceleration independently. The electrostatic actuation reduces temperature dependence and permits *in situ* calibration.

#### TASK SIGNIFICANCE:

The accelerometer will find applications in microgravity research, inertial navigation, seismology, geophysics, planetary physics, and DoD programs. The device under development is optimized for a microgravity application, and additional applications should require only minor modifications.

#### PROGRESS DURING FY 1994:

The micromachined accelerometer is assembled from four parts, and all four parts have been manufactured, bonded together, and tested. The data acquisition electronics has been built and passed thermal, vibration and EMI testing at Lewis Research Center. The control and measurement electronics has been designed and breadboarded in readiness for testing and characterization.

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#### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 10/92    EXPIRATION: 9/95

PROJECT IDENTIFICATION: 93-1

RESPONSIBLE CENTER: JPL

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## II. MSAD Program Tasks — Advanced Technology Development

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### *High Resolution Pressure Transducer and Controller*

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PRINCIPAL INVESTIGATOR: Dr. Ulf E. Israelsson

Jet Propulsion Laboratory (JPL)

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CO-INVESTIGATORS:

Prof. Peter Michelson

Stanford University

Prof. John Lipa

Stanford University

Dr. Martin Barmatz

Jet Propulsion Laboratory (JPL)

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#### TASK OBJECTIVE:

The objective of this project is to develop high resolution pressure transducers and controllers with many orders of magnitude better performance than presently commercially available. The main use of these devices will be for fundamental science investigations in the area of low temperature physics at helium temperatures.

#### TASK DESCRIPTION:

Two types of pressure transducers will be developed for use in the 1 to 10 bar pressure range. The first will utilize a capacitive readout technique and will be capable of operating at any temperature from 300K to 2K. The second will utilize inductive readout by means of a SQUID and will operate at helium temperatures only (2K to 7K). The expected resolution of these transducers are about one part in  $10^{10}$ , and they will dissipate negligible amounts of heat when operated. The pressure controller will be based on similar techniques as the transducers and will be capable of controlling pressures in the 1 to 10 bar range over a limited pressure swing to near one part in  $10^{10}$ .

#### TASK SIGNIFICANCE:

The current capability of the MSAD program in fundamental low temperature physics is limited to controlling and reading out one experimental variable, temperature, to sub-nano kelvin resolution at helium temperatures. By providing a similar capability for a second variable, pressure, many new important flight investigations can be performed. An example would be to test the validity of the Universality hypothesis in the theory of second order phase transitions by performing high resolution measurements of some experimental quantity at several fixed pressures.

#### PROGRESS DURING FY 1994:

An initial version of the capacitive pressure transducer was designed and constructed. It employs a flexible Silicon membrane which is sealed to a helium sample chamber with an Indium seal. Attached to the back side of the membrane is a thin film capacitor plate. A second thin film capacitor plate is located a short distance away on a fixed block of Silicon. Pressure variations in the sample cell will flex the membrane thereby adjusting the relative capacitance of the circuit. For optimum reproducibility in the fabrication and assembly stages, the entire assembly is produced using Silicon micro machining in existing JPL facilities. The assembled transducer was pressure tested up to 10 bar and repeatedly cycled between room temperature and 77K without experiencing any leak problems. The transducer is currently undergoing sensitivity testing using a thermal platform stabilized to nanokelvin temperatures.

A design for the superconducting pressure transducer was produced. Detailed design calculations were performed in order to optimize performance in the 1 to 10 bar range. The design is similar to the capacitive version, except that the flexing portion is a Niobium membrane. Pressure variations will be picked up by the changing distance between the Niobium ground plane and a Silicon disk on which a set of thin film Niobium coils are laid down. The resultant change in inductance will be read out by a commercial SQUID system. The photolithography mask for laying down the coils on the Silicon disk have been procured and fabrication of the Niobium metal housing and diaphragm have been completed. Assembly will be underway shortly.

## II. MSAD Program Tasks — Advanced Technology Development

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 1  
MS Students: 0  
PhD Students: 0

TASK INITIATION: 10/93 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 963-01-04-00

RESPONSIBLE CENTER: JPL

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Surface Light Scattering Instrument*

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PRINCIPAL INVESTIGATOR: Dr. William V. Meyer

Ohio Aerospace Institute

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#### CO-INVESTIGATORS:

Prof. J.A. Mann

Case Western Reserve University

Dr. P. Tin

Case Western Reserve University

R.B. Rogers

NASA Lewis Research Center (LeRC)

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#### TASK OBJECTIVE:

The objective is the development of an instrument capable of detecting fluid surface phenomena (liquid/liquid and liquid/vapor), such as local temperatures and interface temperature gradients, as well as surface tensions and volume viscosity. This development is maturing with design and fabrication of many portions of the new instruments are well underway.

#### TASK DESCRIPTION:

We started with a traditional room-sized surface light scattering instrument. Our instrument has evolved to a state where the vibration sensitive bulk optics' high power, large space requirements in the traditional surface light scattering instrument have been replaced by a low power, compact laser-diode surface light scattering system. This includes a new "cat's eye" optics design that is immune to the sloshing of the surface being examined. This had been a major problem that kept this useful instrument from seeing commercial applications. A fiber optics version of the instrument is well underway; the ability to detect surface tension gradients (and temperature gradients), using a set of acousto-optic modulator crystals placed in series in the fiber optics line, is being implemented.

#### TASK SIGNIFICANCE:

This instrument will allow noninvasive, noncontact measurements of surface tension. From this, one can extrapolate the local surface temperature (compared to bulk surface temperature) of a known clean sample. Viscosity information is also provided. Until we implemented the auto-tracking optics, vibration encountered in a regular laboratory environment made this instrument difficult to use. This means that a commercial version of this instrument will now likely become a reality. Brookhaven Instruments (among others) has already expressed an interest in making this happen, and has designed—and has provided—a custom version of their next-generation correlator with this in mind. Additionally, this instrument will be valuable for reduced-gravity studies of critical phenomena and interface characterization.

Surface tension is an elusive phenomena which controls many individual processes and everyday phenomena. It is the two-dimensional analog of pressure and attempts to maintain the smallest possible surface area. It affects cooking, cosmetics (improved formulation), tertiary oil recovery (20% - 30% more oil can be pumped from the ground), detergents (better wetting of fibers), controlled release and targeted drug delivery (e.g. liposomes, what can transport through the surface), materials processing (e.g. - steel making), etc. The study of surface tension driven phenomena is often masked by gravitational forces which are not present in the reduced gravity environment of a space station or space shuttle. This instrument provides not only a non-invasive measurement of surface tension from which surface temperature can be extracted, it also gives the viscosity information. Viscosity is the internal friction of a fluid (i.e. how thick or thin it is). This affects liquid crystal displays (flat screens) used in computers, etc.

#### PROGRESS DURING FY 1994:

Breadboarded grating systems (used with 680 nm and 780 nm laser diodes) with and without enhanced versions of "cat's eye" optics. Breadboarded and successfully tested new anti-slosh optical train in both transmission and

## II. MSAD Program Tasks — Advanced Technology Development

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reflection modes. Our surface tension measurements with the new optical train were better than 0.1% for transmission mode (with low surface tension fluids: water, ethanol, acetone) and better than 0.5% for reflection mode (with a high surface tension fluid, gallium). Designed and in process of building a completely fiber optic surface light scattering system using polarization maintaining fiber couplers. Adapted surface light scattering instrument to be usable as a heterodyning system, which permits non-invasive measurement of surface tension, viscosity and temperature gradients. Received BI-8050 correlator (built for us by Brookhaven Instruments); using its unique data taking capabilities to analyze data taken with anti-slosh optics. Designed and had fabricated a graded linear transmission filter which we will use in a high-amplitude (1-2 microns) spectrometer. Computer code for thermocapillary ripples on a curved surface is nearly complete. It includes thin surface film effects, including absorption, and it lays the foundation for a generalized theory of the dynamics of interfaces. Adapted laser vibrometer to measure high amplitude ripples on fluid surfaces. Successfully tested vibrometer on Putterman's (UCLA) levitated drop experiment. Reported hardware developments at NASA's 1994 Fluids and Materials Workshop and at SPIE's 1994 International Space Optics Conference.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 1  
PhD Students: 1

TASK INITIATION: 10/91 EXPIRATION: 9/95

PROJECT IDENTIFICATION: 92-4

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Crystal Growth Instrumentation Development: A Protein Crystal Growth Studies Cell*

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PRINCIPAL INVESTIGATOR: Dr. Marc L. Pusey

NASA Marshall Space Flight Center (MSFC)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

The goal is to design and construct prototype cells which will permit study of the growth process of protein crystals by a variety of techniques. Two cell designs are proposed. The first and most important will be for studies of the solution concentration gradients surrounding a growing crystal. The second design will be to study interfacial features of protein crystals under growth and etching conditions. A second goal of the proposed work will be to develop practical methods for protein storage prior to use in crystal growth (and other) experiments. The characteristics desired for a Protein Crystal Growth Analysis Chamber system for  $\mu$ g growth studies are:

1. Several types of growth cells to accommodate specific study goals
2. Temperature control of the growth cell from  $0-40 \pm 0.05$  °C
3. Control of nucleation and growth of crystals at a defined location
4. A fluidic system to accurately prepare crystal growth solutions from stock solutions of protein, precipitant, buffer, etc. and deliver them to the growth cell
5. The cells should be accessible for additions to and/or modification of the solution
6. The ability to do follow-up experiments based upon preceding growth runs
7. Some cell adjustment to bring selected crystal faces in line (perpendicular or parallel) with the optical axes
8. Easy accessibility to other solution measurement systems (pH, conductivity, etc.) for maximum data return
9. Remotely operable from the ground to the maximum extent possible
10. Ability to maintain proteins in a viable state prior to use

#### **TASK DESCRIPTION:**

Current microgravity ( $\mu$ g) protein crystal growth (PCG) hardware systems are hybrid, attempting to both acquire data about the processes of crystallization while growing crystals suitable for x-ray diffraction studies. This leads to compromises in the design, with the result that neither approach is successfully accomplished. The proposed hardware will be designed solely for studying the growth of protein crystals, which requires fewer experimental growth cells, but each having the maximum amount of data return and control of solution physico-chemical parameters.

For the upcoming year's effort the primary emphasis will be shifted from growth cell design to methods suitable for protein storage during long term (3 months)  $\mu$ g missions. This will employ 8-12 different proteins, each stored in two forms (in solution or as a freeze-dried powder) at three different temperatures (20 °C, 4 °C, and -20 °C). At periodic intervals the stored material will be sampled and tested for changes in crystallizability, turbidity, and structural and functional integrity. These measurements will determine if the net charge, molecular size, or shape, of the protein have altered during storage. Concurrently with the above we will begin designing and testing potential storage containers suitable for g use.

#### **TASK SIGNIFICANCE:**

In usage onboard an orbiting platform ground-based researchers will be able to monitor and change selected solution parameters within the growth cells, and conduct follow-on experiments based upon recently acquired data. This will enable the realistic studying of how  $\mu$ g affects the protein crystal growth process, and how it can be improved both in space and on Earth. Long-term protein solution storage and stability will become a dominating concern as the time scale of g experiments increases. Developing methods for long-term protein storage will improve both this and future g-based instrumentation.

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## II. MSAD Program Tasks — Advanced Technology Development

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### PROGRESS DURING FY 1994:

This project is a new start, building upon a previous one year proof-of-concept project. In that project, an initial cell design was found to have flaws, primarily how to replace the solution within the cell without bubbling and the designing of optical systems to pass through a curved glass interface. A second cell design resulted which has a square cross section, a volume of ~4 ml's, and can accommodate a small peltier thermoelectric device for cold spot crystal nucleation. Solution exchange is by flow through the cell. This cell is currently being tested for thermal regulation, and the ability to grow protein crystals using the cold spot. The new cell design can maintain short-term thermal regulation, measured on the surface of the growth cell when filled with liquid, within  $\pm 0.015$  °C with minimal insulation and without the windows installed. Longer term fluctuations over  $\pm 0.05$  °C are primarily due to room temperature changes and the lack of protection from air currents. Power consumption when the heaters are 100% on is 6.25 watts, and ~1 watt to maintain the cell at 12 °C above ambient once the set temperature has been reached.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/94    EXPIRATION: 10/96

PROJECT IDENTIFICATION:    963-70-06

NASA CONTRACT NO.:    In-house

RESPONSIBLE CENTER: MSFC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Multizone Furnace Control Algorithm Development*

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PRINCIPAL INVESTIGATOR: Dr. Bruce N. Rosenthal

NASA Lewis Research Center (LeRC)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

The objective of this ATD is to quantify the performance of a multivariable control system for eventual application to multizone crystal growth furnaces.

Multivariable control techniques are important because they can handle explicit coupling among furnace components rather than treating the coupling dynamics as disturbances. The major justification for use of multivariable furnace control is that thermal interactions exist between furnace heating elements. In a multizone furnace these interactions may be strong enough that accounting for them explicitly by the controller can improve performance. It has been our experience working with multizone furnaces that these interactions exist and they can be significant. Not only do interactions occur between heating elements, but also the ampoule, insulation and outside disturbances contribute to interactions as well. Until now we have not quantified the extent of these interactions and the ability of multivariable control methods to handle the complex nature of thermal interactions expected in multizone furnace systems.

#### **TASK DESCRIPTION:**

Our approach is to quantify multivariable methods developed under prior ATD research by studying the performance of multivariable control on a multizone test article. This will help us understand the controller performance in terms of multizone heating. This research will involve two major tasks. The first task will concern the design and construction of the multizone test article. The second task will concern the application and testing of the controller. The controls aspect of this project will involve the expertise of Professor Celal Batur from the University of Akron.

#### **TASK SIGNIFICANCE:**

Quantification will establish a base-line performance index for use of this technology in future applications.

#### **PROGRESS DURING FY 1994:**

Multizone test article construction has been completed. Specification for the test article are:

- Room temperature to 700 C° operation.
- 10 amp PWM power conditioning per element.
- Type K .005 inch ceramic insulated thermocouples
- 50 Mhz 486 computer control
- 16 bit ADC, 12 bit DAC
- Eight - 3 ohm, 30 volt .25 inch dia. incoloy sheath heating elements.
- Angle iron frame - 9 inch x 9 inch x12 inch outside dimensions. 1/2" thick insulated walls - alumina sheet.

Two interaction indices are being used. Interaction Index 1 ( $II_1$ ) compares areas under the temperature step response curves of each thermocouple. Interaction Index 2 ( $II_2$ ) compares under the derivative of the temperature step response curves of each thermocouple. The advantage of  $II_2$  is it can distinguish between temperature responses involving overshoots and undershoots. Initial comparisons have been made using the distance between the control thermocouples and the heating elements as a variable.  $II_2$  quantified the interaction of the distance variable showing a spreading out of the temperatures as the distance increased. The trend was not as strong when using  $II_1$ .

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## II. MSAD Program Tasks — Advanced Technology Development

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Remaining activities include identifying other variables for quantification including for example furnace wall position, internal wall surface condition, and sample material. The project is scheduled for completion by September, 1994.

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### STUDENTS FUNDED UNDER RESEARCH:

BS Students: 0  
MS Students: 0  
PhD Students: 1

TASK INITIATION: 10/92 EXPIRATION: 10/94

PROJECT IDENTIFICATION: 963-03-0C-00

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *High Resolution Thermometry and Improved SQUID Readout*

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PRINCIPAL INVESTIGATOR: Dr. Peter Shirron

Goddard Space Flight Center

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CO-INVESTIGATORS:

Dr. Michael J. DiPirro

Goddard Space Flight Center

Dr. James G. Tuttle

Hughes/STX

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#### **TASK OBJECTIVE:**

The objective of this work is to develop two technologies which are becoming increasingly central to low temperature microgravity research: high resolution thermometers and Superconducting Quantum Interference Devices (SQUIDs). A penetration depth thermometer (PDT) which uses thin film superconductors is being developed which can meet the performance of existing paramagnetic salt thermometers, while offering greater flexibility and immunity to particle radiation. Advanced SQUID sensors have been manufactured with far better intrinsic energy resolution than commercially available sensors. These sensors use a second stage array of 100-200 SQUIDs to amplify the signal from a single input SQUID. SQUID arrays and control electronics will be developed and evaluated for potential use in spaceflight applications.

#### **TASK DESCRIPTION:**

This work is currently proceeding along two independent paths. First, PDT sensors are being fabricated and evaluated. Different geometries and film deposition techniques for are being explored to determine which factors most significantly impact the sensitivity and noise of the thermometer. Measurements of temperature sensitivity and ultimate resolution will then be made reading out the thermometer with a commercial SQUID system. Second, SQUID arrays which have been obtained from HYPRES, Inc. are being evaluated in terms of noise, power dissipation, and sensitivity to operating temperature and external magnetic fields. Control electronics will be designed, fabricated, and tested with these sensors.

Eventually, a PDT will be fabricated to take advantage of the capabilities of the SQUID arrays which have significantly lower input inductance than commercial SQUIDs. The PDT can either be made smaller and achieve the same resolution or can be coupled through a transformer to achieve better resolution.

#### **TASK SIGNIFICANCE:**

When studying critical phenomena (for example, the lambda point of liquid helium), the focus is usually on how certain parameters such as density or heat capacity change very close to the critical point. It is crucial for such experiments to determine or control the thermodynamic state to very high precision. Particularly in the microgravity environment, the precision to which temperature and/or pressure can be measured sets the limit for science return. Advances in current state-of-the-art for thermometry and SQUID sensors therefore translate into improvements in the quality of microgravity experiments. The SQUID technology will be particularly beneficial since SQUIDs have a wide range of uses, both in spaceflight applications and in ground-based laboratories.

#### **PROGRESS DURING FY 1994:**

1. Very uniform and high quality superconducting cylindrical aluminum films on sapphire mandrels have been fabricated and tested. The films have been tailored in thickness to have superconducting transition temperatures in the range of 1.2 to above 1.8 K, though higher temperatures ( $>2.2\text{K}$ ) are possible. Inductance measurements indicate these films will realize better than the goal of 1 nanoKelvin/ $\approx 88\text{Hz}$  temperature resolution when a commercial dc SQUID is used for readout. Assembly and testing of a thermometer are underway.
2. The initial focus was to determine the sources of noise in our test apparatus for the SQUID arrays. Since the intention is to be able to operate these SQUIDs in relatively unshielded environments, considerable effort was spent distinguishing between noise which is intrinsic to the device and that arising from external sources. Coupled to this

## II. MSAD Program Tasks — Advanced Technology Development

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effort was a determination of optimum operating conditions. Above frequencies of 300 Hz, we have succeeded in demonstrating current resolution of  $1.3 \text{ pA}/\sqrt{88 \text{ Hz}}$ , just shy of the goal of  $1 \text{ pA}/\sqrt{88 \text{ Hz}}$ . This corresponds to an energy resolution of 200 h, more than a factor of four better than commercial SQUIDs. Below 300 Hz the noise power spectrum exhibits a  $1/f$  dependence, rising in amplitude to about  $20 \text{ pA}/\sqrt{88 \text{ Hz}}$  at 1 Hz. The emphasis for further work in this area will be to investigate  $1/f$  noise reduction techniques, such as are employed in commercial systems.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/93    EXPIRATION: 8/96

PROJECT IDENTIFICATION: 963-70-70

RESPONSIBLE CENTER: GSFC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Determination of Soot Volume Fraction Using Laser-Induced Incandescence*

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PRINCIPAL INVESTIGATOR: Dr. Karen J. Weiland

NASA Lewis Research Center, (LeRC)

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CO-INVESTIGATORS:

Dr. Randall Vander Wal

NYMA, Inc.

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#### **TASK OBJECTIVE:**

This project will develop a laser-based optical diagnostic technique, laser-induced incandescence (LII), for the measurement of soot volume fraction. The major objectives are to obtain the hardware and choose a combustion system of interest that yields relatively high soot concentrations, verify and develop the technique in the laboratory, including establishing LII on a quantitative basis, demonstrate LII in a laboratory flame under a variety of conditions and geometries, conduct reduced-gravity tests with LII and other complementary optical measurements, and assess the usefulness of LII as a measure of soot volume fraction.

#### **TASK DESCRIPTION:**

We intend to develop laser-induced incandescence (LII) as a two-dimensional imaging diagnostic for the measurement of soot volume fraction for microgravity combustion research. LII, in conjunction with other optical imaging techniques, would provide unparalleled temporal and spatial resolution, and therefore, yield insight and sensitivity into soot formation and oxidation processes. Present methods of measuring soot volume fraction are limited to line-of-sight methods. These methods offer poor temporal and spatial resolution and require assumptions about the path length and soot physical properties. LII utilizes the spatial and temporal properties of pulsed laser excitation to heat soot to far greater temperatures than ordinary flame temperatures and exploits the resultant blue-shifted incandescent emission from the laser-heated soot. The incandescence is predicted theoretically to be a measure of soot volume fraction, which is the feature of soot that governs many physical processes such as radiative heat transfer from flames. This technique will be extended to two-dimensional imaging applications and will be calibrated against gravimetric sampling.

#### **TASK SIGNIFICANCE:**

Successful development of this diagnostic technique will provide detailed data on soot formation, growth, and oxidation processes for a variety of combustion systems, which may lead to a better understanding and control of sooting processes in flames.

#### **PROGRESS DURING FY 1994:**

Manufacturers were contacted regarding capabilities of their lasers for performing in a reduced-gravity environment. Initially, a premixed flat flame produced on a McKenna burner was chosen for technique verification.

Our primary goal was to determine how to minimize laser-induced and natural interferences, maximize the signal, and ensure a known dependence of the signal upon soot volume fraction. A pulsed Nd:YAG laser provided the excitation light at either 1064 or 532 nm. All technique verification measurements were performed in a premixed ethylene/air flat flame and stabilized by either a chimney or flat plate. For laboratory demonstration measurements, a small coflow burner was chosen. Combustion measurements were performed on heptane and decane droplets supported by an optical fiber.

Spectrally-resolved scans over the range of 300 to 900 nm of the emission from the laser-heated soot for 532 nm excitation were obtained. The data obtained at a low laser intensity with a prompt gate of 50 nsec, show bands at 308 and 342 nm, assignable to OH emissions, which are nonlinearly dependent upon the 532 nm laser intensity. An additional peak at 850 nm was observed at all laser intensities and is attributed to excited oxygen atom emission. Slight changes in the laser beam quality significantly alter the spatial power density, and cause significant variations in the observed photochemical interferences. A delay between the laser pulse and detection allows any molecular and photofragment emission to decay but allows sufficient LII signal to be collected.

## II. MSAD Program Tasks — Advanced Technology Development

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Emission scans using 1064 nm excitation for two different laser pulse power intensities and collection gates lack significant photochemical effects. Although soot absorbs less at 1064 nm than at 532 nm, the power available from the laser is sufficient to heat the soot to incandescent temperatures. For most of the subsequent measurements, 1064 nm light is used.

We measured the measured variation of the signal with laser intensity for two collection wavelengths centered at 425 nm and 550 nm and two different C/O ratios for an excitation wavelength of 1064 nm. A clear saturation regime exists near 100 to 150 MW/cm<sup>2</sup> where the signal is quite independent of the laser power, in excellent agreement with calculations. Similar behavior is found for 532 nm excitation, although at a lower intensity. Operation in this region ensures that slight attenuation of the laser beam due to absorption through the soot field does not affect the observed signal.

The time-resolved behavior of the LII signal detected at 450 nm was studied. The decays at a particular wavelength are phenomenological averages reflecting a range of initial "incandescent" temperatures and time-varying contributions of various cooling processes. The signal obtained at low laser intensity is best fit with a single decay time of  $130 \pm 5$  nsec and is interpreted as the temporal response of the laser-heated soot to the cooling of the particle by a combination of conductive, convective and radiative processes. At high laser intensities the majority of the incandescence nearly tracks the laser pulse. The fit is a sum of two exponential time decays of  $9 \pm 1$  and  $50 \pm 10$  nsec. The rapid temporal response of the LII signal observed at high laser intensity is in good agreement with the response curve calculated by Melton for a single carbon particle of 20 nm diameter irradiated with a laser intensity of 100 MW/cm<sup>2</sup>.

We obtained the dependence of the LII signal on soot volume fraction by comparison to conventional line-of-sight extinction measurements. There is demonstrated linearity between the LII signal and soot volume fraction at two different detection wavelengths. The linearity of the LII signal over an order of magnitude in soot volume fraction produced by the different C/O ratios encourages the use of LII for quantitative measurements of soot volume fraction.

One-dimensional radial profiles were obtained of the soot. Even though visually the flames appear cylindrically uniform at a given axial height, the soot fields are not uniform. The immediate utility of such plots is that one obtains a less ambiguous measure of the path length through the flame as opposed to that from natural flame images. Single laser shot, two-dimensional images were obtained in a coflow diffusion flame, both vertically and horizontally. In both images, the camera detects no scattered light, and no background luminosity from the flame. Sequences of images can be used to build a three-dimensional picture of the soot in the flame.

Images of the natural flame luminosity of a burning decane droplet were obtained from a second camera 260 ms after ignition. The flame is elongated in the opposite direction of the gravitational vector; as a result of natural convection. To the eye, the base of the flame near the lower half of the droplet is blue while the very luminous region above the droplet is yellow. Temporally-resolved LII images were taken of the same test. The displaced location of the soot evolved from the burning droplet flame was located predominantly as a conical section in the wake region above the droplet. Higher camera intensifier gains, which led to saturation of the LII signal in the upper flame region, did not reveal any soot in the bottom portion of the flame.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/93    EXPIRATION: 9/97

PROJECT IDENTIFICATION: 94-2

RESPONSIBLE CENTER: LeRC

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## II. MSAD Program Tasks — Advanced Technology Development

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Multi-Color Holography*

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PRINCIPAL INVESTIGATOR: Mr. William K. Witherow

NASA Marshall Space Flight Center (MSFC)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

A noncontact method of simultaneously determining concentration and temperature variations in fluid systems is underway. An additional benefit will be the additional simultaneous data acquisition capability and thus a possible reduction in the number of experiment runs required per mission.

#### **TASK DESCRIPTION:**

In this system, two fluid parameters will be varied simultaneously, and this technique will measure the variations by using two different frequency lasers.

#### **TASK SIGNIFICANCE:**

More complete multivariable research on fluid science experiments will be enabled by this new capability.

#### **PROGRESS DURING FY 1994:**

Experiments have been performed involving simultaneous concentration and temperature gradients in sugar solutions and salt solutions. Gels have been investigated for possible use so that the concentration gradients can be more stable, but it has been found that even at low concentrations they have such a high optical density that they cannot be used. A breadboard system that can be flown on the KC-135 has been constructed. The system uses a diode laser at 680 nm wavelength and a diode pumped frequency doubled Nd-YAG laser at 532 nm wavelength. The system uses fiber optics to guide the beams to the desired locations. Phase shifts for phase shifting interferometry are induced by a piezo-electric cylinders which have several turns of the fiber optic cables wound around them. A computer system for automated data collection and analysis has been constructed.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/91    EXPIRATION: 9/94

PROJECT IDENTIFICATION: 963-70-01

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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## II. MSAD Program Tasks — Advanced Technology Development

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### *Ceramic Cartridges via Sintering and Vacuum Plasma Spray*

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PRINCIPAL INVESTIGATOR: Mr. Frank R. Zimmerman

NASA Marshall Space Flight Center (MSFC)

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CO-INVESTIGATORS:

No Co-I's Assigned to this Task

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#### **TASK OBJECTIVE:**

The objective of this development work is to provide a manufacturing process for containment cartridges used in high temperature (1200 ° to 2000 °C) crystal growth furnaces. A thermal spray process is being used to build up refractory metals and ceramics into a containment cartridge for high temperature, single crystal semi-conductor growth experiments. This process uses high energy plasma inside a low pressure (100-200 torr) inert environment to apply layers of material onto a removable mandrel. A variety of materials are being characterized and evaluated against a demanding set of requirements, including high service temperature (1700 °C), oxidation resistance, and resistance to liquid metal attack. Techniques to spray form refractory metals (tungsten, molybdenum, niobium, tantalum) and ceramics (alumina, boron nitride) are being developed in the Plasma Spray Cell at Marshall Space Flight Center. These plasma spray formed materials are evaluated for mechanical properties, density, microstructure, and resistance to liquid semiconductor attack.

#### **TASK DESCRIPTION:**

A thermal spray process is being used to build up refractory metals and ceramics into a containment cartridge for high temperature, single crystal semi-conductor growth experiments. This process uses high energy plasma inside a low pressure (100-200 torr) inert environment to apply layers of material onto a removable mandrel. A variety of materials are being characterized and evaluated against a demanding set of requirements, including high service temperature (1700 °C), oxidation resistance, and resistance to liquid metal attack. Techniques to spray form refractory metals (tungsten, molybdenum, niobium, tantalum) and ceramics (alumina, boron nitride) are being developed in the Plasma Spray Cell at Marshall Space Flight Center. These plasma spray formed materials have been evaluated for mechanical properties, density, microstructure, and resistance to liquid metal attack. Forming techniques and the resultant mechanical and metallurgical properties will be presented.

#### **TASK SIGNIFICANCE:**

NASA's Crystal Growth Furnace (CGF) has flown on the United States Microgravity Lab (USML-1) mission to conduct single crystal growth work on a variety of semiconductor materials. These semiconductor crystals (GaAs, CdZnTe, HgZnTe, HgCdTe) are grown in quartz ampoules surrounded by insulation, thermocouples, and other instrumentation. The entire experiment is contained within a Sample Ampoule Cartridge (SAC), as shown in Figure 1, which facilitates handling and installation into the furnace. The SAC also functions as a containment system, which is required to protect the flight crew from the highly toxic materials used in these crystal growth experiments. If the ampoule containing the crystal growth material should rupture, these cartridges provide a necessary level of containment. The containment requirement is complicated by the fact that most of these crystal growth compounds are known to aggressively attack most common structural metals at furnace operating temperatures. Currently, these cartridges are made of materials which are attacked at rate that will not fully penetrate the cartridge within the duration of the experiment. For experiments of longer duration, a more chemically resistant material is required to assure no breach in the containment will occur. Regardless of the material used, the current manufacturing sequence is labor intensive, dependent upon product availability, and contains several fabrication steps and joining processes.

## II. MSAD Program Tasks — Advanced Technology Development

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### PROGRESS DURING FY 1994:

Vacuum plasma spray parameters were developed for a variety of refractory metals and ceramics. These parameters were scaled up to produce over 220 full size (24 in. long by 1.0 in. diam) monolithic cartridges on reusable graphite mandrels.

Compatibility tests were conducted with most of the available refractories in Ga, GaAs, Ge and high temperature furnace environments.

Aluminum oxide and silicide coatings were demonstrated as effective oxidation barriers for protecting the underlying refractory metal.

VPS formed refractory cartridges can meet leak rate and pressure requirements of several crystal growth furnaces under consideration or planned for flight.

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STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 10/93    EXPIRATION: 10/95

PROJECT IDENTIFICATION: 963-70-05

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

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### III. Microgravity Science & Applications Bibliographic Citations for FY 1994

- **Flight Research Tasks**
  - Benchmark Science ..... III-655
  - Biotechnology ..... III-656
  - Combustion Science ..... III-657
  - Fluid Physics ..... III-661
  - Materials Science ..... III-665
- **Ground Research Tasks**
  - Benchmark Science ..... III-671
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- **ATD**
  - Advanced Technology Development ..... III-705

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## Flight: Benchmark Science

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## Appendix

A	Table of Acronyms .....	A-1
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The following list of acronyms, though by no means complete, includes those used in this document as well as some that are often found in text associated with Microgravity Science and Applications research and which may be encountered when reviewing references cited in the bibliography herein.

AADSF .....	Advanced Automated Directional Solidification Furnace
AAL .....	Aero-acoustic Levitation
ACRT .....	Accelerated Crucible Rotation Technique
AO .....	Announcement of Opportunity
AOMS .....	Advanced Optical Monitoring Systems
APCF .....	Advanced Protein Crystallization Facility
APCG .....	Advanced Protein Crystal Growth
ATD .....	Advanced Technology Development
BDPU .....	Bubble, Drop and Particle Unit
BTf .....	Biotechnology Facility
CADAP .....	Computer-Aided Dendrite Analysis Program
CAST .....	Casting and Solidification Technology
CBE .....	Chemical Beam Epitaxy
CCD .....	Charge-coupled Device
CFLSE .....	Critical Fluid Light Scattering Experiment
CFTE .....	Critical Fluid Thermal Equilibration Experiment
CFVME .....	Critical Fluid Viscosity Measurement Experiment
CGF .....	Crystal Growth Furnace
CM-1 .....	Combustion Module
CNES .....	Centre Nationale d'Études Spatiales [The French National Center for Space Studies]
CoDR .....	Conceptual Design Review
CPF .....	Critical Point Facility
CSA .....	Canadian Space Agency
CVD .....	Chemical Vapor Deposition
CVTE .....	Chemical Vapor Transport Experiment
CW .....	Continuous Wave
DARA .....	Deutsche Agentur für Raumfahrtangelegenheiten [German Space Agency]
DARTFire .....	Diffusive and Radiative Transport in Fires
DLR .....	The German Aerospace Research Establishment
DPM .....	Drop Physics Module
DSC .....	Differential Scanning Calorimetry
DSF .....	Directional Solidification Furnace
DTA .....	Differential Thermal Analysis
EDM .....	Engineering Development Model
EDS .....	Energy Dispersive Spectroscopy

EHD .....	Electrohydrodynamic
ESA .....	European Space Agency
FDSMM .....	Fluid Dynamics and Solidification of Metallic Melts
FES .....	Fluids Experiments System
FES/VCGS .....	Fluid Experiment/Vapor Crystal Growth System
FFEU .....	Free-Flow Electrophoresis Unit
FFDF .....	Fluid Physics and Dynamics Facility
GaAs .....	Gallium Arsenide
GAS Can .....	Get-away Special Canister
GBX .....	Glovebox
GGFC .....	Geophysical Fluid Flow Cell
HRT .....	High-Resolution Thermometer
ICE .....	Interface Configuration Experiment
IDGE .....	Isothermal Dendritic Growth Experiment
IML .....	International Microgravity Laboratory
IWG .....	Investigator Working Group
JPL .....	Jet Propulsion Laboratory
JSC .....	Johnson Space Center
KSC .....	Kennedy Space Center
LaRC .....	Langley Research Center
LDV .....	Laser Doppler Velocimetry
LeRC .....	Lewis Research Center
LIF .....	Large Isothermal Furnace
LPE .....	Lambda Point Experiment
MCF .....	Modular Combustion Facility
MEPHISTO .....	Matériel pour l'Étude des Phénomènes Intéressants de la Solidification sur Terre et en Orbite [Interesting Phenomena of Solidification on Earth and in Orbit]
MGM .....	Mechanics of Granular Materials
MMSL .....	Microgravity Materials Science Laboratory
MP .....	Microgravity Pressure
MPA .....	Microgravity Pressure, Ambient
MSA .....	Microgravity Science and Applications (Program)
MSAD .....	Microgravity Science and Applications Division
MSFC .....	Marshall Space Flight Center
MSL .....	Microgravity Science Laboratory
NASA .....	National Aeronautics and Space Administration
NASDA .....	National Space Development Agency of Japan
NIST .....	National Institute for Standards and Technology
NLO .....	Nonlinear Optical

NRA .....	NASA Research Announcement
NRC .....	National Research Council
OARE.....	Orbital Acceleration Research Experiment
OLMSA .....	Office of Life Sciences and Microgravity Applications
OMCVD .....	Organometallic Chemical Vapor Epitaxy
OPCGA .....	Observable Protein Crystal Growth Apparatus
PBE .....	Pool Boiling Experiment
PCG .....	Protein Crystal Growth
PDA .....	Phase Doppler Pneumometer
PIV .....	Particle Image Velocimetry
PMZF .....	Programmable Multizone Furnace
POCC .....	Payload Operations Control Center (at MSFC)
PVT .....	Physical Vapor Transport
RAPIV .....	Radiographic Particle Image Velocimetry
RDR .....	Requirements Definition Review
RPCVD .....	Remote Plasma-enhanced Chemical Vapor Epitaxy
RSD .....	Rainbow Schlieren Deflectometry
RTE .....	Radiative Transfer Equation
QSAM.....	Quasi-steady Acceleraton Measurement System
SAA .....	South Atlantic Anomaly
SAMS .....	Space Acceleration Measurement Systems
SCF .....	Supercritical Fluid
SEM .....	Scanning Electron Microscopy
SL .....	ESA Spacelab (in Space Shuttle cargo bay; e.g., IML and USML)
SQUID .....	Superconducting Quantum Interference Detector
SSCE .....	Solid Surface Combustion Experiment
SSFF .....	Space Station Furnace Facility
STDCE .....	Surface Tension Driven Convection Experiment
STEP .....	Satellite Test of the Equivalence Principle
STS .....	Space Transportation System
SVP .....	Space Vehicle Pressure
TEM .....	Transmission Electron Microscopy
TEMPUS .....	Electromagnetic Containerless Processing Facility
TGDF .....	Turbulent Gasjet Diffusion Flames
TME .....	Test and Measurement Equipment
TMS .....	Thermal Maneuvering System
USML .....	United States Microgravity Laboratory
USMP .....	United States Microgravity Payload
VCGS .....	Vapor Crystal Growth System

VDA .....	Vapor Diffusion Apparatus
VIBES .....	Vibration Isolation in a Box Experiment System
XRD .....	X-ray Diffraction

## Academic: Domestic

W. F. Anderson — Vanderbilt University

E. Arnold — Rutgers University

J. K. Baird — University of Alabama, Huntsville

C. W. Carter, Jr. — University of North Carolina, Chapel Hill

W. J. Cook — University of Alabama, Birmingham

E. W. Czerwinski — University of Texas, Medical Branch

L. J. DeLucas — University of Alabama, Birmingham

G. K. Farber — Pennsylvania State University

J. R. Knox — University of Connecticut

K. L. Krause — University of Houston

O. Lel-Kabbanin — University of Alabama, Birmingham

A. McPherson — University of California, Riverside

E. J. Meehan — University of Alabama, Huntsville

E. F. Meyer — Texas A&M University

T. L. Nagabhushan — Schering-Plough Research Institute

S. Narayana — University of Alabama, Birmingham

R. J. Naumann — University of Alabama, Huntsville

D. H. Ohlendorf — University of Minnesota

W. M. Rosenblum — University of Alabama, Birmingham

V. K. Senadhi — Temple University

L. Sieker — University of Washington

P. B. Sigler — Yale University

C. Smith — University of Alabama, Birmingham

D. Smith — Medical College of Buffalo

M. Sundaralingam — Ohio State University

D. Voel — University of Pennsylvania

W. W. Wilson — Mississippi State University

## **Academic: Foreign**

S. Abara — Kyoto University (Japan)

C. Betzel — European Molecular Biology (Germany)

L. T. Delbaere — University of Saskatchewan (Canada)

G. G. Dodson — University of York (England)

J. Drenth — University of Groningen (The Netherlands)

W. G. Lever — The Australian National University (Australia)

K. Moore — University of Alabama, Birmingham

P. G. Righetti — University di Milano (Italy)

W. Weber — University of Hamburg

D. Yang — McMaster University (Canada)

## **Industry: Domestic**

Y. S. Babu — BioCryst Pharmaceuticals, Inc.

D. J. Duchamp — The Upjohn Company

D. S. Eggleston — Smith Kline Beecham Pharmaceuticals

H. M. Einspahr — Bristol-Meyers Squibb

B. C. Finzel — The Upjohn Company

N. D. Jones — Eli Lilly Research

M. A. Navia — Vertex Pharmaceuticals, Inc.

P. Reichert — Schering-Plow Research Institute

B. H. Rubin — Eastman Kodak Company

F. R. Salemme — 3-Dimensional Pharmaceuticals, Inc.



R. M. Swee — Associated Universities, Inc.

J. Thomson — Vertex Pharmaceuticals, Inc.

P. P. Trotta — Schering-Plough Research Institute

K. D. Watenpaugh — The Upjohn Company

P. C. Weber — Dupont Merck Pharmaceutical Co.

## **Industry: Foreign**

## **Government: Domestic**

D. Carter — NASA Marshall Space Flight Center (MSFC)

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